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A.S.T.M. STANDARDS ADOPTED IN 1925

In the serial designation prefixed to the following titles, the initial letter indicative of the general classification and the first number are permanent. The final number indicates the year of adoption as standard.

B. Non-Ferrous Metals

C. Cement, Lime, Gypsum, and Clay Products

D. Miscellaneous Materials

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AMERICAN SOCIETY

FOR

TESTING MATERIALS

ORGANIZED IN 1898
INCORPORATED IN 1902

A.S.T.M. TENTATIVE STANDARDS

1925

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FOREWORD

This volume contains the 193 tentative specifications, methods of test, definitions of terms and recommended practices in effect at the time of its publication. The term "tentative" applies to a proposed standard published for one or more years with a view of eliciting criticism before it is formally adopted as standard by the Society. The Tentative Standards submitted or revised in any given year appear in the Proceedings for that year. In addition, *all* Tentative Standards in effect in any given year are published collectively in a Book of A.S.T.M. Tentative Standards which is issued each year about October 1, the present volume containing those in effect as of August 28, 1925, the date of canvassing the letter ballot on the adoption of standards authorized at the 1925 annual meeting.

Containing all the Tentative Standards, the Book of Tentative Standards should prove most convenient for reference purposes. The specifications and methods of test, although still in the trial stage of the A.S.T.M. procedure, represent the latest thought of the committees on the subjects covered and therefore are finding important applications in the various industries. Since the tentative standards may frequently be used in conjunction with A.S.T.M. standards, the volume is complementary to the volumes containing the standards, namely, the Book of A.S.T.M. Standards with its supplements.

Under the title of each tentative standard there is printed a note emphasizing its tentative status and the possibility of annual revision, and soliciting criticism and suggestions.

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The term "Tentative Standard" is applied to a proposed Standard which is printed in the Proceedings and Book of A.S.T.M. Tentative Standards for one or more years with a view of eliciting criticism, of which the committee concerned will take due cognizance before recommending final action towards the adoption of such Tentative Standard by formal action of the Society.

In the serial designations prefixed to the following titles, the initial letter indicative of the general classification and the first number are permanent, and are retained when a Tentative Standard is adopted as Standard. The final number indicates the year of original issue, or in the case of revision, the year of last revision. The letter "T" is appended to the designation of all Tentative Standards. Thus, the tentative standards which were originated or revised in 1925 are designated by the final number 25. The serial designations of Tentative Standards which have for any reason not been adopted as standard and have been discontinued, are permanently dropped.

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TENTATIVE REVISIONS OF A.S.T.M. STANDARDS

A. FERROUS METALS

B. NON-FERROUS METALS

C. Cement, Lime, Gypsum, and Clay Products

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TENTATIVE SPECIFICATIONS

FOR

STEEL TIE PLATES.[1]

Serial Designation: A 67 – 20 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1916; REVISED, 1917, 1918, 1920.

Material Covered. 1. These specifications cover two grades of steel tie plates, namely: soft and medium. The soft grade will be used unless otherwise specified.

I. MANUFACTURE.

Process. 2. The steel shall be made by either or both the following processes: Bessemer or open-hearth.

II. CHEMICAL PROPERTIES AND TESTS.

Chemical Composition. 3. The steel shall conform to the following requirements as to chemical composition:

BESSEMER.	SOFT GRADE.	MEDIUM GRADE.
Carbon, per cent..........	not under 0.06	not under 0.12
Phosphorus, per cent......	not over 0.10	not over 0.10
OPEN-HEARTH.		
Carbon, per cent..........	not under 0.12	not under 0.20
Phosphorus, per cent......	not over 0.06	not over 0.06

Ladle Analyses. 4. (*a*) A carbon determination shall be made of each melt of bessemer steel, and two analyses every 24 hours representing the average of the elements carbon, manganese, phosphorus and sulfur contained in the steel, one for each 12-hour period respectively. These analyses shall be made from drillings taken at least $\frac{1}{8}$ in. beneath the surface of a test ingot obtained during the pouring of the melts. The chemical composition thus

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 3.

(*b*) An analysis of each melt of open-hearth steel shall be made by the manufacturer to determine the percentages of carbon, manganese, phosphorus and sulfur. This analysis shall be made from drillings taken at least $\frac{1}{8}$ in. beneath the surface of a test ingot obtained during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 3.

Check Analyses. 5. An analysis may be made by the purchaser from a finished tie plate representing each melt of open-hearth steel, and each melt or lot of 10 tons of Bessemer steel. The carbon content thus determined shall not be less than that specified in Section 3, and the phosphorus content shall not exceed that specified in Section 3 by more than 25 per cent.

III. PHYSICAL PROPERTIES AND TESTS.

Bend Tests. 6. The bend test specimens specified in Section 7 shall bend cold through 180 deg. around a pin the diameter of which is equal to the thickness of the specimen for the soft grade, and to twice the thickness of the specimen for the medium grade, without cracking on the outside of the bent portion.

Test Specimens. 7. Bend test specimens shall be taken from the finished tie plates, or from the rolled bars; and longitudinally with the rolling. They shall be rectangular in section, not less than $\frac{1}{2}$ in. in width between the planed sides, and shall have two parallel faces as rolled. They shall be free from ribs or projections. Where the design of the tie plates is such that the specimen cannot be taken between the ribs or projections, these ribs or projections shall, in preparing the specimen, be planed off even with the main surface of the tie plate.

Optional Bend Tests. 8. If preferred by the manufacturer and approved by the purchaser, the following bend test may be substituted for that described in Section 6:

A piece of the rolled bar shall bend cold through 90 deg. around a pin the diameter of which is equal to the thickness of the section where bent for the soft grade, and to twice the thickness of the section where bent for the medium grade, without cracking on the outside of the bent portion.

9. (*a*) One bend test shall be made from each melt of open-hearth steel, or from each melt or lot of 10 tons of bessemer steel. **Number of Tests.**

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

IV. WORKMANSHIP AND FINISH.

10. The tie plates shall be smoothly rolled, true to template, and shall be straight and out of wind on the surface which will form the bearing for the rail. They shall conform to the dimensions specified by the purchaser, with the following permissible variations: **Workmanship.**

(*a*) For plates with shoulders parallel to the direction of rolling, a variation of $\frac{1}{32}$ in. in thickness, $\frac{1}{8}$ in. in rolled width, and $\frac{3}{16}$ in. in sheared length will be permitted.

(*b*) For plates with shoulders perpendicular to the direction of rolling, a variation of $\frac{1}{32}$ in. in thickness, $\frac{3}{16}$ in. in rolled width, and $\frac{1}{8}$ in. in sheared length will be permitted. The distance from the face of shoulder to the outside end of plate shall not vary more than $\frac{1}{4}$ in., and from the face of shoulder to the inside end not more than $\frac{1}{8}$ in.

11. The finished tie plates shall be free from burrs and other surface deformations caused by the shearing and punching; they shall also be free from other injurious defects and shall have a workmanlike finish. **Finish.**

V. MARKING.

12. The name or brand of the manufacturer, the section and the year of manufacture shall be rolled in raised letters and figures on the outside of the shoulder of the plates, and a portion of this marking shall appear on each finished tie plate. **Marking.**

VI. INSPECTION AND REJECTION.

13. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the tie plates ordered. The manufacturer shall afford the inspector, free of cost, all reasonable facilities to satisfy him that the tie plates are being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the **Inspection.**

place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 14. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 5 shall be reported within five working days from the receipt of samples.

(*b*) Tie plates which show injurious defects subsequent to their acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing. 15. Samples tested in accordance with Section 5, which represent rejected tie plates, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS

FOR

CARBON TOOL STEEL[1]

Serial Designation: A 71–24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standars of the Society and is subject to annual revision.

ISSUED, 1917; REVISED, 1920, 1921, 1924

Material Covered. 1. (*a*) These specifications cover carbon tool steels, the composition of which shall be as agreed upon by the purchaser and vendor, with the permissible variations specified in Section 4.

(*b*) These steels are suitable for lathe and planer tools, drills and milling cutters, dies and other miscellaneous purposes, etc.

I. MANUFACTURE

Process. 2. The steel shall be made either by the crucible or electric furnace process.

Annealing. 3. The material shall be furnished unannealed unless otherwise specified.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 4. The composition of the tool steels shall be as agreed upon by the purchaser and vendor with the following permissible variations in composition:

	Permissible Variation, per cent	
	Over	Under
Carbon	0.05	0.05
Manganese, when under 1.00	0.10	0.10
Manganese, when over 1.00	0.20	0.20
Phosphorus	0.005	....
Sulfur	0.005	....
Silicon	0.15	0.15

Other elements as agreed.

Method of Sampling. 5. (*a*) Milled samples for analysis shall be taken from the sample bar to represent the full cross-section, or drillings shall be taken by drilling a bar midway between the center and the surface, parallel to the axis of the bar. In either case the decarburized material shall be rejected.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

(*b*) In general, the minimum depth to which the surface shall be removed before a sample is taken shall be in accordance with the following requirements:

Thickness or Diameter, in.	Material Removed, in.
1 or under	$\frac{1}{32}$
Over 1 to $2\frac{1}{2}$, inclusive	$\frac{1}{16}$
Over $2\frac{1}{2}$ to 4, inclusive	$\frac{1}{8}$
Larger sizes	As agreed

(*c*) In the case of drill rod and other material of similar finis where freedom from decarburization is specified, samples shall be taken from the surface in addition to the methods described in Pargraph (*a*).

Standard Methods of Analysis. 6. In case of dispute, the chemical analysis shall be made in accordance with the Standard Methods of Chemical Analysis of Plain Carbon Steel (Serial Designation: A 33) of the American Society for Testing Materials.[1]

III. MANUFACTURING TOLERANCES IN DIMENSIONS

7. Variations in the cross-sectional dimensions of the material shall not exceed the following amounts:

Forged and Rolled Material

	Permissible Variation, in.	
	Over	Under
$\frac{1}{2}$ in. or under	0.010 ($\frac{1}{100}$)	0.010 ($\frac{1}{100}$)
Over $\frac{1}{2}$ in. to 1 in., inclusive	0.015 ($\frac{1}{64}$)	0.015 ($\frac{1}{64}$)
Over 1 in. to $1\frac{1}{2}$ in., inclusive	0.031 ($\frac{1}{32}$)	0.031 ($\frac{1}{32}$)
Over $1\frac{1}{2}$ in. to 2 in., inclusive	0.046 ($\frac{3}{64}$)	0.031 ($\frac{1}{32}$)
Over 2 in. to 4 in., inclusive	0.125 ($\frac{1}{8}$)	0.031 ($\frac{1}{32}$)
Over 4 in. to 6 in., inclusive	0.156 ($\frac{5}{32}$)	0.031 ($\frac{1}{32}$)
Over 6 in. to 8 in., inclusive	0.188 ($\frac{3}{16}$)	0.063 ($\frac{1}{16}$)
Over 8 in.	0.250 ($\frac{1}{4}$)	0.063 ($\frac{1}{16}$)

Finished Material (Drill Rod)

$\frac{7}{16}$ in. and under	0.0005	0.0005
Over $\frac{7}{16}$ in.	0.001	0.001

IV. FINISH

Finish. 8. (*a*) The material shall be free from injurious defects and shall have a workmanlike finish.

(*b*) All drill rods shall be coated with a rust preventive.

V. MARKING

Marking. 9. (*a*) Identification marks shall be stamped on the material as agreed by the vendor and the purchaser. All identification marks shall be about $\frac{1}{4}$ in. in height when the size permits.

[1] 1924 Book of A.S.T.M. Standards.

(*b*) In lieu of marking as specified in Paragraph (*a*), the ends of bars and rods may be painted as agreed by the vendor and the purchaser.

Bundling Small Sizes. 10. All rods and bars of which the largest cross-sectional dimension is $\frac{3}{8}$ in. or less shall be boxed or bundled, each box or bundle to contain bars or rods of the same size, shape and class of material. A metal tag shall be securely attached to the wrapping wire or box. Rods and bars of cross-sectional dimensions exceeding $\frac{3}{8}$ in. and not exceeding $1\frac{1}{2}$ in., where convenient to vendor and purchaser, may be bundled and marked as specified above. Bundles shall be of suitable size to permit easy handling and shall be securely fastened.

VI. INSPECTION AND REJECTION

Inspection. 11. The vendor shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications.

Comparison with Selective Sample. 12. The steel shall correspond in quality and characteristics to the samples submitted under the specifications and approved by the purchaser.

Rejection. 13. Material which does not conform to the chemical composition and specified dimensional variations, or which at any time shows injurious defects inherent in the steel, will be rejected and the vendor hall be notified.

APPENDIX

TENTATIVE RECOMMENDED PRACTICE IN THE HEAT TREATMENT OF PLAIN CARBON TOOL STEEL[1]

General.—This specification covers the process to be followed in the heat treating of plain carbon tool steel.

PART I.—NORMALIZING OF TOOL STEEL BEFORE HARDENING

Process.—Operations:
- Heating.
- Cooling.

Heating: Place steel into furnace so as to expose maximum surface area. Heat uniformly to a temperature above the upper critical point indicated in Table I, and hold at this temperature for sufficient time to obtain complete penetration of heat and refinement of grain.

Cooling: Remove from furnace and cool freely in air.

[1] This is a tentative Recommended Practice approved by the Standards Committee of the American Society for Steel Treating and is published by permission of that Society.

TABLE I.—NORMALIZING

Carbon Range, per cent	Normalizing Temperature, deg. Fahr.	Normalizing Temperature, deg. Cent.
0.65 – 0.80	1475 – 1525	800 – 830
0.80 – 0.95	1475 – 1500	800 – 815
0.95 – 1.10	1500 – 1575	815 – 860
1.10 – 1.25	1575 – 1650	860 – 900

CROSS SECTIONS, WEIGHT AND TIME

Thickness of Largest Section of Unit	Weight of Unit, lb. (Approximate)	Approximate Time of Heating, hr.	Approximate Time of Soaking, hr.
Up to and including 1 in.	Up to 1000	$\frac{3}{4}$	$\frac{1}{2}$
Over 1 in. and including 2 in.	Over 100 and including 300	$1\frac{1}{4}$	$\frac{1}{2}$
Over 2 in. and including 3 in.	Over 300 and including 500	$1\frac{3}{4}$	$\frac{3}{4}$
Over 3 in. and including 4 in.	Over 500 and including 1000	$2\frac{1}{4}$	1
Over 4 in. and including 5 in.	Over 1000 and including 1500	$2\frac{3}{4}$	1
Over 5 in. and including 8 in.	Over 1500 and including 2000	$3\frac{1}{2}$	$1\frac{1}{2}$

PART II.—HEAT TREATING OF PLAIN CARBON TOOL STEEL

Process.—Operations:

Heating for quenching.
Quenching.
Tempering or drawing.

Heating: Heat the steel uniformly to the temperature indicated in Table II.

Quenching: Quench from this temperature in water, but do not cool below temperature of boiling water (212° F.).

TABLE II.—HEAT TREATING.

Carbon Range, per cent	Hardening Temperature, deg. Fahr.	Quenching Medium and Its Temperature
0.65 – 0.80	1550 – 1450	Water at 70° F.
0.81 – 0.95	1460 – 1410	Water at 70° F.
0.96 – 1.10	1390 – 1430	Water at 70° F.
1.11 – 1.25	1380 – 1420	Water at 70° F.

Tempering or Drawing: Reheat immediately in oil or salt ($NaNO_3$) or furnace for the time and at the temperature specified in Table III and cool.

TABLE III.—TEMPERING ON DRAWING TOOL STEEL

Results Desired	Tempering Medium	Temperature, deg. Fahr.
Relieving Strains	Oil	350 to 375
Strain Relieving and Reduction of Brittleness	Oil	400 to 500
To Relieve Strains and Toughen	Oil	500 to 600

NOTES ABOUT THE PROCESS

1. *General.*—The recommended practice for the heat treatment of tool steel applies to highest quality performance of tools for general purposes only. For specific applications where special structural requirements seem to be necessary, deviation from the recommended practice must be left to the judgment of the individual heat treater or metallurgist.

2. *Normalizing.*—A normalizing treatment for all tool steels is recommended to obtain a uniform and refined grain structure, which enables the operator to predict the behavior and performance of the steel tool during heating and quenching for hardening.

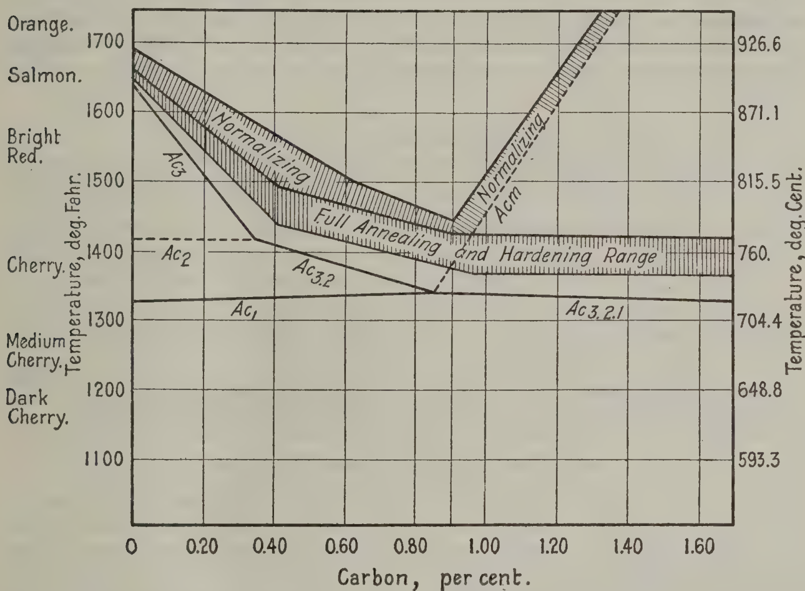

FIG. 1.—Critical Range Diagram for Iron-Carbon Alloys, showing Normalizing, Annealing and Hardening Ranges.

The variation in temperature for the different carbon ranges becomes evident upon a careful examination of the critical range diagram. A temperature very much in excess of that required to produce solid solution (austentite) is conducive of austenitic grain growth and intergranular weakness.

A low normalizing temperature for high-carbon steels (1.25 to 1.50 per cent carbon) which fails to break up the massive cementite results in a brittle structure, since the rate of diffusion of the excess cementite depends upon the temperature. The solubility of carbide in iron being greater, or increases as the temperature rises, the rate of this solubility is equivalent to the slope of the Acm. line in the diagram.

The higher normalizing temperatures given in Table I apply to the lower carbon ranges and the lower temperature to the higher carbon ranges for steel of the hypo-eutectoid composition, or, in other words, "the normalizing temper-

atures for hypo-eutectoid steels varies indirectly as the carbon percentage rises or falls, and the normalizing temperature of hyper-eutectoid steels varies directly with the percentage of carbon."

3. *Heating for Quenching.*—(*a*) The wide range of temperature in the 0.65 to 0.80 per cent carbon steel is needed, as a number of tools such as shear blades, arbors, mandrels, and others are all around the low point, and, because of mass and forms, require somewhat higher temperatures for quenching.

(*b*) The quenching temperatures given are at the lowest temperature range consistent with highest quality tools; deviations from it are not recommended, but may be practical for diverse reasons.

Quenching.—Water is the universal quenching medium, and by varying its temperature and manner of application for the abstraction of heat, almost any degree of variation of structural conditions of the tool steel can be obtained. There are, however, special cases where oil may be a more suitable quenching medium.

CARBON STEELS SUITABLE FOR VARIOUS USES

Carbon Content 0.65 to 0.85 Per Cent.—Shear blades, boiler snaps and cups, hammers, stamping and pressing dies, mining drills.

Carbon Content 0.81 to 0.95 Per Cent.—Hot and cold sets, chisels, dies, shear blades, mining drills, smiths' tools, set hammers, swages, flatteners.

Carbon Content 0.96 to 1.10 Per Cent.—Small cold chisels, hot sets, small shear blades, large pinchers, large taps, granite drills, trimming dies, turning tools, planer tools, drills, cutters, slotting and milling tools, mill picks, circular cutters, small shear blades, threading dies.

Carbon Content 1.11 to 1.25 Per Cent.—Small cutters, small taps, drills, slotting and planing tools, wood-cutting tools, turning tools, razors, etc.

SUITABLE TEMPERING HEATS FOR VARIOUS TOOLS

Temperatures, 350 to 390° F.—Lathe tools for brass and copper alloys. Milling cutters for brass and copper alloys. Scraper and cutting tools for soft metals and micarta. Drawing mandrels, drawing dies, bone-cutting tools, hammer-faces, steel engraving tools, wood-carving tools, cutting tools for iron and steel, hand tools, threading dies for brass.

Temperature, 400 to 500° F.—Hand taps and dies, hand reamers, drills, bits, cutting dies, penknives, milling cutters, chasers, inserted sawteeth, press dies for sheet steel, rock drills, taps and dies, wire-drawing dies, dental and surgical instruments, twist drills.

Temperature, 500 to 600° F.—Bending and forming dies, shear blades, chuck jaws, forging dies, drifts, gages, press-dies, flat drills, reamers, chisels and tools for wood-cutting, hammers and drop dies, axes, lathe tools for copper augers, cold chisels, coppersmith tools, grinders, screw drivers, molding and planing tools, hacksaws, needles, butcher knives, saws and tools.

TENTATIVE SPECIFICATIONS

FOR

HIGH-SPEED TOOL STEEL[1]

Serial Designation: A 92 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924

Material Covered.

1. (*a*) These specifications cover high-speed tool steels, the composition of which shall be as agreed upon by the purchaser and vendor, with the permissible variations specified in Section 4.

(*b*) These tool steels are suitable for lathe and planer tools, milling cutters, drills, etc.

I. MANUFACTURE

Process.

2. The tool steel shall be made either by the crucible or electric furnace process.

Annealing and Hardness.

3. The steel shall be furnished annealed unless otherwise specified. The microstructure, Brinell hardness and fracture tests shall be as agreed upon between the vendor and the purchaser.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition

4. The composition of the tool steels shall be as agreed upon by the purchaser and vendor with the following permissible variations in composition:

	Permissible Variations, per cent	
	Over	Under
Carbon	0.05	0.05
Manganese	0.15	0.15
Phosphorus	0.01	....
Sulfur	0.01	....
Silicon	0.15	0.15
Chromium	0.50	0.50
Vanadium	0.25	0.25
Tungsten	0.75	0.75
Cobalt	0.50	0.50
Other elements as agreed.		

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

Method of Sampling.

5. (*a*) Milled samples for analysis shall be taken from the sample bar to represent the full cross-section, or drillings shall be taken by drilling a bar midway between the center and the surface, parallel to the axis of the bar. In either case the decarburized material shall be rejected.

(*b*) In general, the minimum depth to which the surface shall be removed before a sample is taken shall be in accordance with the following requirements:

Thickness or Diameter, in.	Material Removed, in.
1 or under	$\frac{1}{32}$
Over 1 to $2\frac{1}{2}$, inclusive	$\frac{1}{16}$
Over $2\frac{1}{2}$ to 4, inclusive	$\frac{1}{8}$
Larger sizes	As agreed

(*c*) In the case of drill rod and other material of similar finish where freedom from decarburization is specified, samples shall be taken from the surface in addition to the methods described in Paragraph (*a*).

Standard Methods of Analysis.

6. In case of dispute, the chemical analysis shall be made in accordance with the Standard Methods of Chemical Analysis of Alloy Steels (Serial Designation: A 55) of the American Society for Testing Materials.[1]

III. MANUFACTURING TOLERANCES IN DIMENSIONS

Manufacturing Tolerances in Dimensions.

7. Variations in cross-sectional dimensions of the material shall not exceed the following amounts:

	Permissible Variations, in. Over	Under
Forged and Rolled Material		
$\frac{1}{2}$ in. or under	0.010 ($\frac{1}{100}$)	0.010 ($\frac{1}{100}$)
Over $\frac{1}{2}$ in. to 1 in., inclusive	0.015 ($\frac{1}{64}$)	0.015 ($\frac{1}{64}$)
Over 1 in. to $1\frac{1}{2}$ in., inclusive	0.031 ($\frac{1}{32}$)	0.031 ($\frac{1}{32}$)
Over $1\frac{1}{2}$ in. to 2 in., inclusive	0.046 ($\frac{3}{64}$)	0.031 ($\frac{1}{32}$)
Over 2 in. to 4 in., inclusive	0.125 ($\frac{1}{8}$)	0.031 ($\frac{1}{32}$)
Over 4 in. to 6 in., inclusive	0.156 ($\frac{5}{32}$)	0.031 ($\frac{1}{32}$)
Over 6 in. to 8 in., inclusive	0.188 ($\frac{3}{16}$)	0.063 ($\frac{1}{16}$)
Over 8 in.	0.250 ($\frac{1}{4}$)	0.063 ($\frac{1}{16}$)
Finished Material (Drill Rod)		
$\frac{7}{16}$ in. and under	0.0005	0.0005
Over $\frac{7}{16}$ in.	0.001	0.001

IV. FINISH

Finish.

8. (*a*) The material shall be free from injurious defects and shall have a workmanlike finish.

(*b*) All drill rods shall be coated with a rust preventive.

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[1] 1924 Book of A.S.T.M. Standards.

V. MARKING

9. (*a*) Identification marks shall be stamped on the material as agreed by the vendor and the purchaser. All identification marks shall be about $\frac{1}{4}$ in. in height when the size permits. **Marking.**

(*b*) In lieu of marking as specified in Paragraph (*a*), the ends of the bars and rods may be painted as agreed by the vendor and the purchaser.

10. All rods and bars of which the largest cross-sectional dimension is $\frac{3}{8}$ in. or less shall be boxed or bundled, each box or bundle to contain bars or rods of the same size, shape and class of material. A metal tag shall be securely attached to the wrapping wire or box. Rods and bars of cross-sectional dimensions exceeding $\frac{3}{8}$ in. and not exceeding $1\frac{1}{2}$ in., where convenient to vendor and purchaser, may be bundled and marked as specified above. Bundles shall be of suitable size to permit easy handling and shall be securely fastened. **Bundling Small Sizes.**

VI. INSPECTION AND REJECTION

11. The vendor shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. **Inspection.**

12. The steel shall correspond in quality and characteristics to the samples submitted under the specifications and approved by the purchaser. **Comparison with Selective Sample.**

13. Material which does not conform to the chemical composition and specified dimensional variations, or which at any time shows injurious defects inherent in the steel, will be rejected and the vendor shall be notified. **Rejection.**

APPENDIX

TENTATIVE RECOMMENDED PRACTICE IN THE HEAT TREATMENT OF 18-PER-CENT TUNGSTEN HIGH-SPEED STEEL[1]

General.—This specification covers the process to be followed in heat treating high-speed steel.

Process.—Operations:

1. Heating for annealing.
2. Cooling.
3. Heating for hardening—(*a*) Preheating—(*b*) Heating for quenching.
4. Quenching.
5. Drawing for secondary hardness.

[1] This is a tentative Recommended Practice approved by the Standards Committee of the American Society for Steel Treating and is published by permission of that Society.

1. *Heating for Annealing.*—Heat slowly and uniformly to a temperature of 1600° F. (871° C.) and hold for complete refinement.

2. *Cooling.*—Cool in furnace, infusorial earth, mica, lime or any medium, that will permit of uniform slow cooling.

3. *Heating for Hardening.*—(*a*) Preheating—Heat slowly and uniformly to 1500° F. (816° C.) in a furnace of sufficient size.

(*b*) Heating for Quenching—Transfer preheated steel to a high-temperature furnace that is maintained at a temperature of 2250° F. to 2400° F. (1232 to 1315° C.), depending on the type of tools being hardened.

In order to obtain the most satisfactory "red hardness" conditions, the steel should be brought rapidly to the higher temperature; but in many cases the character of the cutting edges of certain form tools, such as milling cutters, threading tools, etc., makes it inadvisable to use the higher temperatures owing to destruction of the delicate edges through blistering, pitting, etc. It is, therefore, usual to use the higher temperatures for tools such as rough lathe tools, while the finer class of tools is hardened at the lower temperatures.

High-speed steel tools should not be held at the high heat longer than necessary, since holding at the high hardening temperatures required causes excessive grain growth, with subsequent brittleness of the hardened tools.

Tools that cannot be ground after hardening are often heated in a barium chloride, or some such suitable bath.

4. *Quenching.*—Quench the steel in oil, or air, from the hardening temperature. It is advisable to maintain the oil quenching bath at a temperature of 150 to 200° F. (65 to 93° C.) to eliminate possibility of breakage with intricately shaped tools.

5. *Drawing for Secondary Hardness.*—Reheat uniformly in an open furnace, or preferably in a salt ($NaNO_3$) or lead bath, to 1050 to 1150° F. (565 to 631° C.) for a sufficient length of time, and cool in air, or oil, as preferred.

NOTES ABOUT THE PROCESS

Cooling from Annealing.—Cooling in air should not be permitted, since air cooling from the annealing temperature is apt to result in partial hardening of the tool.

Heating for Hardening.—It is the customary practice to always preheat for hardening in an open furnace, since preheating in a salt bath causes the salt to adhere to the tool, and the subsequent high temperature treatment causes unusual corroding from the adhering salt. Even preheating in lead is objectionable from small quantities of adhering lead. There is not much advantage to be gained in using a molten bath for the preheating, since the preheating temperature may vary over the comparatively wide range of 1400 to 1600° F., and an open furnace is invariably used in practice.

Quenching from Drawing Temperatures.—This should be optional with the steel treater, as quenching from the drawing temperature does not give increased toughness over samples that have air cooled. Evidently the increased toughness resulting when carbon or chrome-vanadium steels are quenched from drawing temperatures of 700 to 1000° F. does not apply to high-speed steels, as impact tests on oil-cooled and air-cooled samples show no difference in favor of the oil-cooled pieces.

TENTATIVE SPECIFICATIONS
FOR
LOW-CARBON-STEEL TRACK BOLTS.[1]

Serial Designation: A 76 – 20 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1918; REVISED, 1920.

I. MANUFACTURE.

Process.

1. (*a*) The steel shall be made by either or both the following processes: Bessemer or open-hearth.

(*b*) The threads may be cut or rolled.

II. CHEMICAL PROPERTIES AND TESTS.

Chemical Composition.

2. (*a*) The steel for the bolts shall conform to the following requirements as to chemical composition:

Phosphorus	Bessemer................	not over 0.10 per cent.
	Open-hearth.............	" " 0.05 "

(*b*) The steel for the nuts shall be "soft steel."

Ladle Analyses.

3. (*a*) A carbon determination shall be made of each melt of Bessemer steel, and two analyses every 24 hours representing the average of the elements carbon, manganese, phosphorus and sulfur, contained in the steel, one for each 12-hour period, respectively. These analyses shall be made from drillings taken at least $\frac{1}{8}$ in. beneath the surface of a test ingot obtained during the pouring of the melts. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 2.

(*b*) An analysis of each melt of open-hearth steel shall be made by the manufacturer to determine the percentages of carbon, manganese, phosphorus and sulfur. This analysis shall be made from drillings taken at least $\frac{1}{8}$ in. beneath the surface of a test ingot obtained during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 2.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

Check Analyses. 4. An analysis may be made by the purchaser from a finished bolt representing each melt. The phosphorus content thus determined shall not exceed that specified in Section 2 by more than 25 per cent.

III. PHYSICAL PROPERTIES AND TESTS.

Tension Tests. 5. The stock from which the bolts are made shall conform to the following requirements as to tensile properties:

Tensile strength, lb. per sq. in	55 000 – 70 000
Elongation in 8 in., min., per cent..........	$\dfrac{1\,500\,000}{\text{Tens. str.}}$

Bend Tests. 6. The test specimen shall bend cold through 180 deg. flat on itself without cracking on the outside of the bent portion.

Test Specimens. 7. Tension and bend test specimens shall be taken from the full-size rolled bars. Tension test specimens shall be of 8-in. gage length.

Number of Tests. 8. (*a*) One tension and one bend test shall be made from each melt of steel. When the melts can not be identified, one tension and one bend test shall be made from each lot of 10 tons or fraction thereof.

(*b*) If any test specimen develops flaws, it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 5 and any part of the fracture is outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Retests. 9. If the results of the physical tests of any test lot do not conform to the requirements specified, two additional tension and two additional bend tests shall be made from such lot, all of which shall conform to the requirements specified.

IV. PERMISSIBLE VARIATIONS.

Permissible Variations. 10. The bolts and nuts shall conform to the dimensions specified by the purchaser subject to the following variations: A variation of $\frac{1}{32}$ in. under and $\frac{1}{64}$ in. over the specified diameter of the shank of the bolt will be permitted. The diameter of rolled thread shall not exceed the diameter of the shank more than $\frac{1}{16}$ in. for bolts $\frac{7}{8}$ in. in diameter and under, nor more than $\frac{3}{32}$ in. for bolts 1 in. in diameter and over. The outside diameter of the finished rolled threads shall not exceed the diameter of

the shank of a cut thread bolt of corresponding size. The length of the bolt under the head shall not vary more than $\frac{1}{8}$ in. from that specified. A variation in the dimensions of the elliptical shoulders under the head of $\frac{1}{32}$ in. and a taper of the shoulder of $\frac{1}{32}$ in. will be permitted.

V. WORKMANSHIP AND FINISH.

11. The bolts and nuts shall be neatly formed and free from fins or nicking. The head of the bolt shall be concentric with and firmly joined to the shank, with the under side at right angles to the axis of the bolt. The threads shall be sharp and true to gage and of the design specified by the purchaser. The nuts shall have a hand free fit on the bolt from two to six turns, and wrench tight the balance of the screw length without distorting the threads or twisting the shank. They shall be screwed on the bolts before packing, a sufficient number of turns to hold them in place until used. **Workmanship.**

12. The finished bolts and nuts shall be free from injurious defects and shall have a workmanlike finish. **Finish.**

VI. MARKING.

13. (*a*) A letter or brand indicating the manufacturer shall be pressed on the head of the bolt when it is formed. **Marking.**

(*b*) Bolts shall be packed in serviceable packages. All packages shall be plainly marked as to material, size of bolts and name of manufacturer.

VII. INSPECTION AND REJECTION.

14. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the bolts and nuts ordered. The manufacturer shall afford the inspector, free of cost, all reasonable facilities to satisfy him that the bolts and nuts are being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, and shall be so conducted as not to interfere unnecessarily with the operation of the works. **Inspection.**

15. Bolts and nuts which show injurious defects subsequent to their acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified. **Rejection.**

TENTATIVE SPECIFICATIONS

FOR

ELECTRIC CAST-STEEL ANCHOR CHAIN.[1]

Serial Designation: A 77 – 20 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1918; REVISED, 1920.

Material Covered. 1. These specifications cover the cast steel to be used in all parts of the complete chain, including stud links, enlarged links, end links, shackles and shackle pins for anchor chain $1\frac{3}{8}$ in. and over.

I. MANUFACTURE.

Process. 2. (*a*) The steel shall be made by the electric furnace process, and all castings shall be produced in dry sand molds or cores.

(*b*) Each complete shot of chain shall be heat-treated in such manner that the treatment will be complete and uniform throughout its entire length.

Weight. 3. The weight of each shot of chain shall not be less than the minimum weight specified in Table I.

II. PHYSICAL TESTS.

Test Chains. 4. (*a*) Three links shall be selected by the inspector representing the purchaser from any part of each 15 fathoms of chain or portion thereof, and cut out for testing purposes. Such links shall be marked for identification and heat-treated with the length they represent.

(*b*) Such test chains shall become the basis, as provided in Section 5, for the acceptance or rejection of the material entering into the shot of chain they represent.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

Breaking Tests. 5. (*a*) One of the test chains shall be pulled in an approved static testing machine to a breaking load in accordance with the loads specified in Table II. If this first test chain withstands the required tensile breaking load, the shot of chain which it represents shall be accepted so far as this test is concerned.

(*b*) If any link in the first test chain fails to withstand the required tensile breaking load, the second test chain shall be subjected to the same test. Prior to this retest the shot of chain and the second test chain may be subjected to a re-heat treatment at the option of the manufacturer. If the second test chain passes the required test the shot of chain which it represents shall be accepted so far as this test is concerned. If the second test chain fails to pass the required test the shot of chain shall be rejected.

TABLE I.—MINIMUM WEIGHT PER SHOT OF 15 FATHOMS.

Size of Chain, in.	Weight, lb.	Size of Chain, in.	Weight, lb.	Size of Chain, in.	Weight, lb.
$1\frac{3}{8}$	1 625	$2\frac{1}{8}$	3 785	$2\frac{7}{8}$	7 015
$1\frac{7}{16}$	1 775	$2\frac{3}{16}$	4 015	$2\frac{15}{16}$	7 330
$1\frac{1}{2}$	1 935	$2\frac{1}{4}$	4 245	3	7 650
$1\frac{9}{16}$	2 090	$2\frac{5}{16}$	4 485	$3\frac{1}{16}$	7 980
$1\frac{5}{8}$	2 235	$2\frac{3}{8}$	4 725	$3\frac{1}{8}$	8 320
$1\frac{11}{16}$	2 410	$2\frac{7}{16}$	4 960	$3\frac{3}{16}$	8 660
$1\frac{3}{4}$	2 590	$2\frac{1}{2}$	5 265	$3\frac{1}{4}$	9 010
$1\frac{13}{16}$	2 785	$2\frac{9}{16}$	5 535	$3\frac{5}{16}$	9 360
$1\frac{7}{8}$	2 975	$2\frac{5}{8}$	5 815	$3\frac{3}{8}$	9 725
$1\frac{15}{16}$	3 175	$2\frac{11}{16}$	6 105	$3\frac{7}{16}$	10 095
2	3 355	$2\frac{3}{4}$	6 405	$3\frac{1}{2}$	10 475
$2\frac{1}{16}$	3 570	$2\frac{13}{16}$	6 705	$3\frac{3}{4}$	12 025

Proof Tests. 6. (*a*) The complete shot of chain shall then be subjected to a proof test, which shall consist of anchoring each end thereof in an approved static testing machine and subjecting it to the proof load shown in Table II, according to its respective size. If the shot of chain passes this test it shall be accepted.

(*b*) If any link or links, in any one shot of 15 fathoms, fail during the proof test a new link or links may be cast in and the shot re-treated and again subjected to the proof tests. If any link fails on this retest the shot shall be rejected.

Shock Tests. 7. In addition to the breaking and proof tests specified in Sections 5 and 6, the material entering into the manufacture of

the chain shall be subjected to such shock or bend tests as shall be agreed upon between the manufacturer and the purchaser, in order to determine its shock-resisting qualities.

Use of Second Test Chain.

8. In the event that only one test chain is subjected to test, the remaining test chain may be united with and become a part of another complete shot of chain.

III. DIMENSIONS AND PERMISSIBLE VARIATIONS.

Dimensions.

9. All castings shall conform to the dimensions and permissible variations agreed upon between the manufacturer and the purchaser.

TABLE II.—PROOF AND BREAKING LOADS.

Size of Chain, in.	Proof Test, lb.	Breaking Test lb.	Size ot Chain, in.	Proof Test, lb.	Breaking Test, lb.	Size of Chain, in.	Proof Test, lb.	Breaking Test, lb.
$1\frac{3}{8}$	114 000	160 000	$2\frac{1}{8}$	255 000	357 000	$2\frac{7}{8}$	431 500	604 000
$1\frac{7}{16}$ ...	124 500	175 000	$2\frac{3}{16}$	270 000	378 000	$2\frac{15}{16}$	444 500	622 000
$1\frac{1}{2}$	131 500	184 000	$2\frac{1}{4}$	285 000	400 000	3........	457 000	640 000
$1\frac{9}{16}$ ...	137 500	193 000	$2\frac{5}{16}$	302 000	423 000	$3\frac{1}{16}$	469 500	658 000
$1\frac{5}{8}$	149 000	208 000	$2\frac{3}{8}$	318 500	446 000	$3\frac{1}{8}$	482 000	676 000
$1\frac{11}{16}$ ...	160 500	225 000	$2\frac{7}{16}$	335 000	470 000	$3\frac{3}{16}$	494 500	693 000
$1\frac{3}{4}$	173 000	242 000	$2\frac{1}{2}$	353 000	494 000	$3\frac{1}{4}$	506 500	710 000
$1\frac{13}{16}$ ...	185 500	260 000	$2\frac{9}{16}$	366 000	512 000	$3\frac{5}{16}$	518 500	726 000
$1\frac{7}{8}$	198 000	278 000	$2\frac{5}{8}$	379 000	531 000	$3\frac{3}{8}$	530 500	742 000
$1\frac{15}{16}$ ...	211 500	296 000	$2\frac{11}{16}$	392 500	550 000	$3\frac{7}{16}$	541 500	758 000
2.......	226 000	316 000	$2\frac{3}{4}$	405 500	568 000	$3\frac{1}{2}$	553 000	775 000
$2\frac{1}{16}$ ...	240 000	336 000	$2\frac{13}{16}$	418 500	586 000	$3\frac{3}{4}$	588 500	824 000

IV. FINISH.

Finish.

10. All castings shall be free from shrinkage, porosity or other injurious defects which would affect the strength of the chain, and shall have a workmanlike finish.

V. MARKING.

Marking.

11. Each link shall be legibly stenciled with its heat number. Each shot of chain accepted shall be legibly stenciled in accordance with the requirements of the purchaser.

VI. INSPECTION.

12. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the chain ordered. The manufacturer shall afford the inspector, free of cost, all reasonable facilities to satisfy him that the chain is being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment, and shall be so conducted as not to interfere unnecessarily with the operation of the works. **Inspection.**

TENTATIVE SPECIFICATIONS

FOR

STEEL PLATES OF STRUCTURAL QUALITY FOR FORGE WELDING.[1]

Serial Designation: A 78 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1919; REVISED, 1920, 1921, 1923.

Material Covered. 1. These specifications cover steel plates of structural quality suitable for forge welding without the addition of fluxes.

I. MANUFACTURE.

Process. 2. The steel shall be made by the open-hearth process.

II. CHEMICAL PROPERTIES AND TESTS.

Chemical Composition. 3. (*a*) The steel shall conform to the following requirements as to chemical composition:

		Grade A.	Grade B.
Carbon	for plates ¾ in. or under in thickness	not over 0.15 per cent	not over 0.20 per cent
	for plates over ¾ in. in thickness	" 0.17 "	" 0.22 "
Manganese		0.35 to 0.60 "	0.35 to 0.60 "
Phosphorus		not over 0.06 "	not over 0.06 "
Sulfur		" 0.05 "	" 0.05 "

(*b*) The composition of the steel should preferably be free from silicon, nickel or chromium. Where these elements are present the maximum quantity of any one shall not exceed 0.05 per cent.

Ladle Analyses. 4. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of carbon, manganese, phosphorus and sulfur. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 3.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

5. An analysis may be made by the purchaser from a broken tension test specimen representing each melt. The chemical composition thus determined shall conform to the requirements specified in Section 3. **Check Analyses.**

III. PHYSICAL PROPERTIES AND TESTS.

6. (*a*) The material shall conform to the following minimum requirements as to tensile properties: **Tension Tests.**

	GRADE A.	GRADE B.
Tensile strength, lb. per sq. in.	45,000	50,000
Yield point, lb. per sq. in.	0.5 tens. str.	0.5 tens. str.
but in no case less than	24,000	27,000
Elongation in 8 in., per cent	28	25

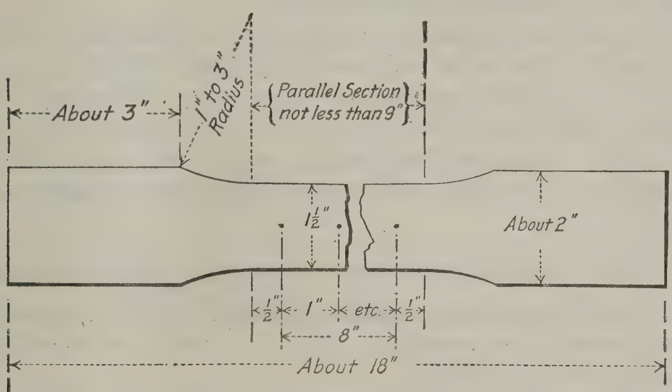

FIG. 1.

(*b*) The yield point shall be determined by the drop of the beam of the testing machine.

7. (*a*) For material over $\frac{3}{4}$ in. in thickness, a deduction from the percentages of elongation specified in Section 6 (*a*) of 0.25 per cent shall be made for each increase of $\frac{1}{32}$ in. of the specified thickness above $\frac{3}{4}$ in., to a minimum of 20 per cent. **Modifications in Elongation.**

(*b*) For material under $\frac{5}{16}$ in. in thickness a deduction from the percentages of elongation specified in Section 6 (*a*) of 1.25 per cent shall be made for each decrease of $\frac{1}{32}$ in. of the specified thickness below $\frac{5}{16}$ in.

8. The test specimen shall withstand being bent cold through 180 deg. flat on itself without cracking on the outside of the bent portion. **Bend Tests.**

9. (*a*) Test specimens shall be prepared for testing from the material in its rolled condition. **Test Specimens.**

(*b*) Test specimens shall be taken longitudinally and, except as specified in Paragraph (*c*), shall be of the full thickness of material as rolled. They may be machined to the form and dimensions shown in Fig. 1, or with both edges parallel.

(*c*) Test specimens for plates over $1\frac{1}{2}$ in. in thickness may be machined to a thickness or diameter of at least $\frac{3}{4}$ in. for a length of at least 9 in.

(*d*) The machined sides of rectangular bend test specimens may have the corners rounded to a radius not over $\frac{1}{16}$ in.

Number of Tests.

10. (*a*) One tension and one bend test shall be made from each melt; except that if material from one melt differs $\frac{3}{8}$ in. or more in thickness, one tension and one bend test shall be made from both the thickest and the thinnest material rolled.

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 6 (*a*) and any part of the fracture is outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

IV. PERMISSIBLE VARIATIONS IN WEIGHT AND THICKNESS.

Permissible Variation.

11. The cross-section or weight of plates shall be covered by the following permissible variations. One cubic inch of rolled steel is assumed to weigh 0.2833 lb.

(*a*) *When Ordered to Weight per Square Foot:* The weight of each lot[1] in each shipment shall not vary from the weight ordered more than the amount given in Table I.

(*b*) *When Ordered to Thickness:* The thickness of each plate shall not vary more than 0.01 in. under that ordered.

The overweight of each lot[1] in each shipment shall not exceed the amount given in Table II.

V. FINISH.

Finish.

12. The finished material shall be free from injurious defects and shall have a workmanlike finish.

VI. MARKING.

Marking.

13. The name or brand of the manufacturer and the melt number shall be legibly rolled or stamped on all finished material. The melt number shall be legibly marked, by stamping if practicable, on each test specimen.

[1] The term "lot" as applied to Table I means all of the plates of each group width and group weight; as applied to Table II, it means all of the plates of each group width and group thickness.

VII. INSPECTION AND REJECTION.

Inspection. 14. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these

TABLE I.—PERMISSIBLE VARIATIONS OF PLATES ORDERED TO WEIGHT.

Ordered Weight, lb. per sq. ft.	Permissible Variations in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Ordered Weights.																		Ordered Weight, lb. per sq. ft.
	Under 48 in.		48 to 60 in., excl.		60 to 72 in., excl.		72 to 84 in., excl.		84 to 96 in., excl.		96 to 108 in., excl.		108 to 120 in., excl.		120 to 132 in., excl.		132 in. or over.		
	Over.	Under.	Over.	Under.	Over.	Under.	Over.	Under.	Over.	Under.	Over.	Under.	Over.	Under.	Over.	Under.	Over.	Under.	
Under 5........	5	3	5.5	3	6	3	7	3	...	...	...	...	...	...	...	...	...	...	Under 5
5 to 7.5 excl..	4.5	3	5	3	5.5	3	6	3	...	...	...	...	...	...	...	...	...	...	5 to 7.5 excl.
7.5 " 10 " .	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	...	...	...	...	7.5 " 10 "
10 " 12.5 " .	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	9	3	10 " 12.5 "
12.5 " 15 " .	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	12.5 " 15 "
15 " 17.5 " .	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	15 " 17.5 "
17.5 " 20 " .	2.5	2	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	17.5 " 20 "
20 " 25 " .	2	2	2.5	2	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	20 " 25 "
25 " 30 " .	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	4.5	3	5	3	25 " 30 "
30 " 40 " .	2	2	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	4.5	3	30 " 40 "
40 or over......	2	2	2	2	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	40 or over

NOTE.—The weight per square foot of individual plates shall not vary from the ordered weight by more than $1\frac{1}{3}$ times the amount given in this table.

TABLE II.—PERMISSIBLE OVERWEIGHTS OF PLATES ORDERED TO THICKNESS.

Ordered Thickness, in.	Permissible Excess in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Nominal Weights.									Ordered Thickness, in.
	Under 48 in.	48 to 60 in., excl.	60 to 72 in., excl.	72 to 84 in., excl.	84 to 96 in., excl.	96 to 108 in., excl.	108 to 120 in., excl.	120 to 132 in., excl.	132 in. or over.	
Under $\frac{1}{8}$............	9	10	12	14	..	..	..	..	..	Under $\frac{1}{8}$
$\frac{1}{8}$ to $\frac{3}{16}$ excl........	8	9	10	12	..	..	..	..	..	$\frac{1}{8}$ to $\frac{3}{16}$ excl.
$\frac{3}{16}$ " $\frac{1}{4}$ "	7	8	9	10	12	..	..	..	..	$\frac{3}{16}$ " $\frac{1}{4}$ "
$\frac{1}{4}$ " $\frac{5}{16}$ "	6	7	8	9	10	12	14	16	19	$\frac{1}{4}$ " $\frac{5}{16}$ "
$\frac{5}{16}$ " $\frac{3}{8}$ "	5	6	7	8	9	10	12	14	17	$\frac{5}{16}$ " $\frac{3}{8}$ "
$\frac{3}{8}$ " $\frac{7}{16}$ "	4.5	5	6	7	8	9	10	12	15	$\frac{3}{8}$ " $\frac{7}{16}$ "
$\frac{7}{16}$ " $\frac{1}{2}$ "	4	4.5	5	6	7	8	9	10	13	$\frac{7}{16}$ " $\frac{1}{2}$ "
$\frac{1}{2}$ " $\frac{5}{8}$ "	3.5	4	4.5	5	6	7	8	9	11	$\frac{1}{2}$ " $\frac{5}{8}$ "
$\frac{5}{8}$ " $\frac{3}{4}$ "	3	3.5	4	4.5	5	6	7	8	9	$\frac{5}{8}$ " $\frac{3}{4}$ "
$\frac{3}{4}$ " 1 "	2.5	3	3.5	4	4.5	5	6	7	8	$\frac{3}{4}$ " 1 "
1 or over...........	2.5	2.5	3	3.5	4	4.5	5	6	7	1 or over

specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 15. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 5 shall be reported within five working days from the receipt of samples.

(*b*) Material which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing. 16. Samples tested in accordance with Section 5, which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
STEEL PLATES OF FLANGE QUALITY FOR FORGE WELDING[1]

Serial Designation: A 89 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923.

Material Covered. 1. These specifications cover steel plates of flange quality suitable or forge welding without the addition of fluxes.

I. MANUFACTURE

Process. 2. The steel shall be made by the open-hearth process.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 3. (*a*) The steel shall conform to the following requirements as to chemical composition:

		Grade A	Grade B
Carbon	for plates $\frac{3}{4}$ in. or under in thickness	not over 0.15 per cent	not over 0.20 per cent
	for plates over $\frac{3}{4}$ in. in thickness	" " 0.17 "	" " 0.22 "
Manganese		0.35 to 0.60 "	0.35 to 0.60 "
Phosphorus	Acid	not over 0.06 "	not over 0.06 "
	Basic	" " 0.04 "	" " 0.04 "
Sulfur		" " 0.05 "	" " 0.05 "

(*b*) The composition of the steel should preferably be free from silicon, nickel or chromium. Where these elements are present the maximum quantity of any one shall not exceed 0.05 per cent.

Ladle Analyses. 4. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of carbon, manganese, phosphorus and sulfur. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 3.

Check Analyses. 5. An analysis may be made by the purchaser from a broken tension test specimen representing each plate as rolled. The chemical composition thus determined shall conform to the requirements specified in Section 3.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

III. PHYSICAL PROPERTIES AND TESTS.

Tension Tests.

6. (*a*) The material shall conform to the following minimum requirements as to tensile properties.

	GRADE A.	GRADE B.
Tensile strength, lb. per sq. in......	45,000	50,000
Yield point, lb. per sq. in..........	0.5 tens. str.	0.5 tens. str.
but in no case less than.......	24,000	27,000
Elongation in 8 in., per cent.......	30	27

(*b*) The yield point shall be determined by the drop of the beam of the testing machine.

Modifications in Elongation.

7. (*a*) For material over $\frac{3}{4}$ in. in thickness, a deduction from the percentages of elongation specified in Section 6 (*a*) of 0.125 per cent shall be made for each increase of $\frac{1}{32}$ in. of the specified thickness above $\frac{3}{4}$ in.

(*b*) For material under $\frac{5}{16}$ in. in thickness, a deduction from the percentage of elongation in 8 in. specified in Section 6 (*a*) of 1.25 per cent shall be made for each decrease of $\frac{1}{32}$ in. of the specified thickness below $\frac{5}{16}$ in.

Bend Tests.

8. The test specimen shall withstand being bent cold through 180 deg. without cracking on the outside of the bent portion, as follows: For material 1 in. or under in thickness, around a pin the diameter of which is equal to the thickness of the specimen; and for material over 1 in. in thickness, around a pin the diameter of which is equal to twice the thickness of the specimen.

Test Specimens.

9. (*a*) Tension test specimens shall be taken longitudinally from the bottom of the finished rolled material, and bend test specimens shall be taken transversely from the middle of the top of the finished rolled material. The longitudinal test specimens shall be taken in the direction of the longitudinal axis of the ingot, and the transverse test specimens at right angles to that axis.

(*b*) Tension and bend test specimens shall be of the full thickness of material as rolled, and shall be machined to the form and dimensions shown in Fig. 1; except that bend test specimens may be machined with both edges parallel.

(*c*) Test specimens for plates over $1\frac{1}{2}$ in. in thickness may be machined to a thickness or diameter of at least $\frac{3}{4}$ in. for a length of at least 9 in.

(*d*) The machined sides of rectangular bend test specimens may have the corners rounded to a radius not over $\frac{1}{16}$ in.

Number of Tests.

10. (*a*) One tension and one bend test shall be made from each plate as rolled.

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 6 (*a*) and any part of the fracture is outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

IV. PERMISSIBLE VARIATIONS IN WEIGHT AND THICKNESS.

11. The thickness of each plate shall not vary more than 0.01 in. under that ordered. **Permissible Variations.**

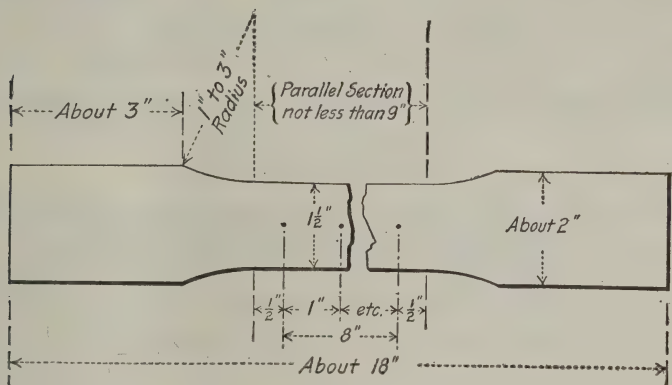

FIG. 1.

The overweight of each lot[1] in each shipment shall not exceed the amount given in Table I. One cubic inch of rolled steel is assumed to weigh 0.2833 lb.

V. FINISH.

12. The finished material shall be free from injurious defects and shall have a workmanlike finish. **Finish.**

VI. MARKING.

13. (*a*) The name or brand of the manufacturer, melt or slab number, class, and lowest tensile strength for the grade specified in Section 6 (*a*) shall be legibly stamped on each plate. The melt or slab number shall be legibly stamped on each test specimen. **Marking.**

(*b*) When specified on the order, plates shall be match-marked as defined in Paragraph (*c*) so that the test specimens representing them may be identified. When more than one plate is sheared from a single slab or ingot, each shall be match-marked so that they may all be identified with the test specimen representing them.

[1] The term "lot" applied to Table I means all of the plates of each group width and group thickness.

(*c*) Each match mark shall consist of two over-lapping circles each not less than $1\frac{1}{2}$ in. in diameter, placed upon the shear lines, and made by separate impressions of a single-circle steel die.

(*d*) Match-marked coupons shall match with the sheets represented and only those which match properly shall be accepted.

VII. INSPECTION AND REJECTION.

Inspection. 14. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

TABLE I.—PERMISSIBLE OVERWEIGHTS OF PLATES ORDERED TO THICKNESS.

Ordered Thickness, in.	Permissible Excess in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Nominal Weights.									Ordered Thickness, in.
	Under 48 in.	48 to 60 in., excl.	60 to 72 in., excl.	72 to 84 in. excl.	84 to 96 in., excl.	96 to 108 in., excl.	108 to 120 in., excl.	120 to 132 in., excl.	132 in. or over	
Under $\frac{1}{8}$	9	10	12	14	..	..	..	..	..	Under $\frac{1}{8}$
$\frac{1}{8}$ to $\frac{3}{16}$ excl	8	9	10	12	..	..	..	..	..	$\frac{1}{8}$ to $\frac{3}{16}$ excl.
$\frac{3}{16}$ " $\frac{1}{4}$ "	7	8	9	10	12	..	..	..	..	$\frac{3}{16}$ " $\frac{1}{4}$ "
$\frac{1}{4}$ " $\frac{5}{16}$ "	6	7	8	9	10	12	14	16	19	$\frac{1}{4}$ " $\frac{5}{16}$ "
$\frac{5}{16}$ " $\frac{3}{8}$ "	5	6	7	8	9	10	12	14	17	$\frac{5}{16}$ " $\frac{3}{8}$ "
$\frac{3}{8}$ " $\frac{7}{16}$ "	4.5	5	6	7	8	9	10	12	15	$\frac{3}{8}$ " $\frac{7}{16}$ "
$\frac{7}{16}$ " $\frac{1}{2}$ "	4	4.5	5	6	7	8	9	10	13	$\frac{7}{16}$ " $\frac{1}{2}$ "
$\frac{1}{2}$ " $\frac{5}{8}$ "	3.5	4	4.5	5	6	7	8	9	11	$\frac{1}{2}$ " $\frac{5}{8}$ "
$\frac{5}{8}$ " $\frac{3}{4}$ "	3	3.5	4	4.5	5	6	7	8	9	$\frac{5}{8}$ " $\frac{3}{4}$ "
$\frac{3}{4}$ " 1 "	2.5	3	3.5	4	4.5	5	6	7	8	$\frac{3}{4}$ " 1 "
1 or over	2.5	2.5	3	3.5	4	4.5	5	6	7	1 or over

Rejection. 15. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 5 shall be reported within five working days from the receipt of samples.

(*b*) Material which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing. 16. Samples tested in accordance with Section 5, which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS

FOR

COLD-DRAWN STEEL WIRE FOR CONCRETE REINFORCEMENT[1]

Serial Designation: A 82–21 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921

1. These specifications cover cold-drawn steel wire to be used as such, or in fabricated form, for the reinforcement of concrete, in gages not less than 0.080 in. nor greater than 0.500 in. **Material Covered.**

2. When wire is ordered by gage number the following relation between number and diameter, in inches, shall apply unless otherwise specified: **Basis of Purchase.**

Gage Number	Equivalent Diameter, in.	Gage Number	Equivalent Diameter, in.
0000000	0.4900	5	0.2070
000000	0.4615	6	0.1920
00000	0.4305	7	0.1770
0000	0.3938	8	0.1620
000	0.3625	9	0.1483
00	0.3310	10	0.1350
0	0.3065	11	0.1205
1	0.2830	12	0.1055
2	0.2625	13	0.0915
3	0.2437	14	0.0800
4	0.2253		

I. MANUFACTURE

3. (*a*) The steel shall be made by either or both the following processes: Bessemer or open hearth. **Process.**

(*b*) The wire shall be cold drawn from rods hot-rolled from billets. **Drawing.**

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

II. PHYSICAL PROPERTIES AND TESTS

Tension Tests. 4. (*a*) The wire, except as specified in Paragraphs (*b*) and (*c*), shall conform to the following minimum requirements as to tensile properties:

Tensile strength, lb. per sq. in.	80,000
Reduction of area, per cent	30

(*b*) For wire to be used in the fabrication of mesh a minimum tensile strength of 70,000 lb. per sq. in. shall be permitted.

(*c*) For wire testing over 100,000 lb. per sq. in. tensile strength, the reduction of area shall be not less than 25 per cent.

Bend Tests. 5. The test specimen shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion, as follows:

For wire 0.3 in. in diameter or under, around a pin the diameter of which is equal to the diameter of the specimen.

For wire over 0.3 in. in diameter, around a pin the diameter of which is equal to twice the diameter of the specimen.

Test Specimens. 6. Tension and bend test specimens shall be of the full-size section of the wire as drawn.

Number of Tests. 7. (*a*) One tension and one bend test shall be made for each 10 tons or less of each size of wire.

(*b*) If any test specimen shows defects or develops flaws, it may be discarded and another specimen substituted.

III. PERMISSIBLE VARIATIONS IN GAGE

8. The diameter of the wire shall not vary more than 0.003 in. from the size ordered.

IV. FINISH

Finish. 9. The finished wire shall be free from injurious defects and shall have a workmanlike finish with smooth surface.

V. INSPECTION AND REJECTION

Inspection. 10. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the wire is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 11. Wire which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

TENTATIVE SPECIFICATIONS

FOR

CARBON-STEEL CASTINGS FOR RAILROADS.[1]

Serial Designation: A 87 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922.

Scope.

1. (*a*) These specifications cover all carbon-steel castings for locomotive and car equipment, and for miscellaneous use, classified as Grade A and Grade B.

(*b*) The purposes for which the two grades are generally used are:

Grade A, unannealed, covers only such special castings as may be approved by the purchaser. *Grade A*, annealed, covers such castings not covered by Grade A, unannealed.

Grade B covers all castings for high stresses such as truck side frames, bolsters, couplers and coupler parts, locomotive frames and locomotive driving and trailer wheel centers.

Basis of Purchase.

2. Grade A castings shall be furnished annealed unless otherwise specified by the purchaser.

Grade B castings shall be furnished annealed.

I. MANUFACTURE.

Process.

3. The steel may be made by one or more of the following processes: open-hearth, electric furnace, crucible or side blow converter.

Annealing.

4. (*a*) The castings shall be allowed to become cool. They shall then be uniformly heated to the proper temperature to refine the grain, and allowed to cool uniformly.

Annealing Lugs.

(*b*) For the purpose of determining the quality of annealing of all castings weighing 150 lb. or over, and of such lighter castings as shall be required by the purchaser or his representative, there shall be provided for removal by the inspector after annealing and also after reannealing, if necessary, at least one and not more than two annealing lugs. The location of the annealing lugs shall be such that

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

when removed by the inspector they shall be indicative of the character of annealing. The standard annealing lug shall be 1 in. in height and 1 in. in width and $\frac{5}{8}$ in. in thickness where it joins the casting.

(*c*) If, in the opinion of the inspector, a casting is not properly annealed, he may at his option require the casting to be re-annealed.

II. CHEMICAL PROPERTIES AND TESTS.

Chemical Composition. 5. The steel shall conform to the following requirements as to chemical composition:

	Grade A.	Grade B.
Manganese, per cent, not over	0.85	0.85
Phosphorus, per cent, not over	0.05	0.05
Sulfur, per cent, not over	0.05	0.05

Ladle Analyses. 6. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of carbon, manganese, phosphorus, sulfur and silicon. This analysis shall be made from drillings taken at least $\frac{1}{4}$ in. beneath the surface of a test ingot obtained during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, when requested, and shall conform to the requirements specified in Section 5.

Check Analyses. 7. An analysis may be made by the purchaser from the broken tension test specimen or from a casting representing each melt. The chemical composition thus determined shall conform to the requirements specified in Section 5. Drillings for analysis shall be taken not less than $\frac{1}{4}$ in. beneath the surface, and shall be taken in such a manner as not to impair the usefulness of a casting.

III. PHYSICAL PROPERTIES AND TESTS.

Tension Tests. 8. (*a*) The steel shall conform to the following minimum requirements as to tensile properties:

	Grade A. Unannealed.	Grade A. Annealed.	Grade B. Annealed
Yield point, lb. per sq. in.	29 250	29 250	36 000
Elongation in 2 in., per cent	$\frac{1\,450\,000}{\text{Tens. str.}}$	$\frac{1\,600\,000}{\text{Tens. str.}}$	$\frac{1\,600\,000}{\text{Tens. str.}}$
but in no case less than	22	24	22
Reduction of area, per cent	$\frac{2\,250\,000}{\text{Tens. str.}}$	$\frac{2\,600\,000}{\text{Tens. str.}}$	$\frac{2\,500\,000}{\text{Tens. str.}}$
but in no case less than	30	35	30

(*b*) The tensile strength shall be reported as information.

(*c*) The yield point shall be determined by the drop of the beam or by the dividers, the method being optional with the inspector, and

at a cross-head speed not to exceed $\frac{1}{8}$ in. per minute. The tensile strength shall be determined at a speed not to exceed $1\frac{1}{2}$ in. per minute.

Test Specimens.

9. (*a*) A sufficient number of test coupons, from which the required test specimen may be prepared, shall be cast attached in the neighborhood of each end of each locomotive frame, attached to each locomotive cylinder, to each wheel center, and to miscellaneous castings weighing over 150 lb. These test coupons shall remain attached to the castings throughout the annealing and until the castings are presented for inspection. If the design of the casting is such that the test coupons cannot be attached, they shall be cast in runners outside of the casting, but attached to it, to represent each melt. The location of the test coupons, as well as the method of casting such coupons, shall be subject to mutual agreement between the inspector and the manufacturer. In the case of any orders for

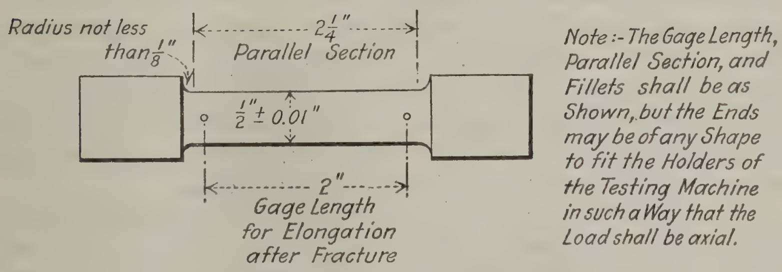

FIG. 1.

castings weighing under 150 lb., the physical properties as required in Section 8, may be determined from an extra or spare test bar cast with and attached to some other casting from the same melt.

(*b*) When sufficient coupons have not been cast, a test specimen may be cut from a finished casting at a location mutually agreed upon by the inspector and the manufacturer.

(*c*) Tension test specimens shall conform to dimensions shown in Fig. 1. The ends shall be not less than $\frac{7}{8}$ in. in diameter and of a length and form to fit the holders of the testing machine in such a way that the load shall be axial.

Number of Tests.

10. (*a*) One tension test shall be made from the neighborhood of each end of each locomotive frame and both tests shall meet the requirements of the specifications. One tension test may be made from each wheel center and each locomotive cylinder casting, but of such castings at least one of each kind in each melt shall be tested. For miscellaneous castings from melts which do not include frames, wheel

centers or cylinders, one tension test shall be made from each melt, except as provided in Section 11 (*a*).

(*b*) If the test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 8 and any part of the fracture is more than $\frac{3}{4}$ in. from the center of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

(*d*) No part of these specifications shall operate to cause any one tension test to apply to more than 40 tons of castings as offered for inspection.

Number of Tests, Consecutive Melts of Miscellaneous Castings.

11. (*a*) After 15 consecutive melts, which may contain any or all classes of castings (except frames, wheel centers, and cylinders) covered by these specifications on one or more orders have been tested and accepted in accordance with the above requirements, the manufacturer may group the succeeding melts in lots of five melts each, but each lot not to exceed 40 tons, the entire group to be accepted if the test specimen selected from the lot fulfills the chemical and physical requirements herein specified. If this test fails, a rehearing will be granted on the melt that the failed bar represents, and the other four melts of the group shall be tested individually.

(*b*) If there is a period of more than six months between shipments of the class of castings covered by these specifications, then each melt shall be tested individually until 15 consecutive melts have been accepted, after which the melts may again be grouped as in Paragraph (*a*).

(*c*) If one or more melts are rejected, each succeeding melt shall be tested individually until 15 consecutive melts have been accepted, after which melts may again be grouped as in Paragraph (*a*).

Number of Tests, Small Orders.

12. In case of small orders for bolsters, truck sides, draft arms, yokes or castings weighing over 150 lb. where the size of the order and the available pattern and foundry equipment are such that not more than five castings can be cast in any one melt, the physical properties, as required in Section 8, will be determined from an extra or spare test coupon cast with and attached to some other casting of the same melt.

Retests.

13. If the results of the physical test lot do not conform to the requirements specified, the manufacturer may re-anneal such lot not more than twice and retest shall be as specified in Section 8.

Alternative Tests to Destruction.

14. In the case of orders including only castings not exceeding 150 lb. in weight, a test to destruction of one casting from each 100

castings or smaller lot may be substituted for the tension tests, at the option of the inspector. This test shall show the material to be ductile, free from injurious defects, and suitable for the purpose intended.

IV. WORKMANSHIP AND FINISH.

Workmanship

15. The castings shall substantially conform to the sizes and shapes shown on the purchaser's drawings, and shall be made in a workmanlike manner.

Finish.

16. (*a*) The castings shall be free from injurious defects.

(*b*) Minor defects which do not impair the strength of the castings may, with the approval of the inspector, be welded by an approved process. The defects shall first be cleaned out to solid metal; and after welding, the castings shall be annealed, if required by the inspector.

V. MARKING.

Marking.

17. The manufacturer's name or indentification mark and the specified pattern number shall be cast on all castings. In addition, the month and year when made shall be cast on all bolsters, truck sides, frames, wheel centers, cylinders, and similar castings. The location and size of numbers shall be agreed upon by the manufacturer and the inspector. In accordance with the standard practice of the individual foundry to identify individual castings, a serial number may be cast or the melt number may be stamped on bolsters, truck sides, frames, wheel centers, cylinders and similar castings as agreed upon by the manufacturer and the inspector. The melt number shall be legibly stamped on all other castings weighing over 150 lb.

VI. INSPECTION AND REJECTION.

Inspection.

18. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of castings ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the castings are being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection.

19. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 7 shall be reported within five working days from the receipt of samples.

(*b*) Castings which show injurious defects subsequent to their acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing. 20. Samples tested in accordance with Section 7, which represent rejected castings, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time

TENTATIVE SPECIFICATIONS

FOR

CARBON-STEEL CAR AND TENDER AXLES[1]

Serial Designation: A 21 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

1. These specifications cover tapered axles up to and including those $6\frac{1}{2}$ in. in nominal diameter at the center. Axles over $6\frac{1}{2}$ in. in diameter at the center shall be purchased under the Standard Specifications for Carbon-Steel Forgings for Locomotives (Serial Designation: A 20) of the American Society for Testing Materials.[2] **Scope.**

I. MANUFACTURE

2. The steel shall be made by the open-hearth or electric-furnace process. **Process.**

II. CHEMICAL PROPERTIES AND TESTS

3. The steel shall conform to the following requirements as to chemical composition: **Chemical Composition.**

Carbon	0.40 – 0.55	per cent
Manganese	0.40 – 0.70	"
Phosphorus, not over	0.05	"
Sulfur, not over	0.06	"

4. An analysis shall be made by the manufacturer from a test ingot taken during the pouring of each melt, to determine the percentage of carbon, manganese, phosphorus, sulfur and, when specified, silicon. Drillings for analysis shall be taken not less than $\frac{1}{4}$ in. beneath the surface of the test ingot. A copy of this analysis shall be given to the purchaser or his representative. This analysis shall conform to the requirements specified in Section 3. **Ladle Analyses.**

5. Analyses may be made by the purchaser from one axle representing each melt. The chemical composition thus determined shall conform to the requirements specified in Section 3. The drillings for these analyses shall be from one end of the test axle at any point midway between the center and surface, using a $\frac{5}{8}$-in. drill. **Check Analyses.**

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

These Tentative Specifications, when adopted as standard, will supersede the present Standard Specifications for Carbon-Steel Car and Tender Axles (Serial Designation: A 21 – 18), 1924 Book of A.S.T.M. Standards.

[2] 1924 Book of A.S.T.M. Standards.

III. PHYSICAL PROPERTIES AND TESTS

Drop Tests. 6. (*a*) The test axle shall be so placed on supports 3 ft. apart, that the tup will strike it midway between the ends. It shall stand without fracture five blows from a tup of 2240 lb. falling from a height H, such that H in feet equals the square of the diameter of the axle at the center in inches, $H=d^2$. The axle shall be turned through 180 deg. after the first and third blows.

(*b*) Before an axle is drop tested, the center shall be calipered to the nearest $\frac{1}{8}$ in., and the height of drop in feet, to the nearest $\frac{1}{2}$ ft., shall not be less than the square of the actual diameter, at the center, in inches.

(*c*) The permanent set produced by the first blow shall not exceed that given by the following formulas, in which L=length of axle in inches, and d=diameter of axle at center in inches.

For axles over 65 in. in length:

$$\left(\frac{L}{1.9d} - \frac{d}{2}\right) + \tfrac{1}{2} \text{ in} \qquad (1)$$

For axles 65 in. or under in length:

$$\left(\frac{L}{1.9d} - \frac{d}{2}\right) + 1 \text{ in} \qquad (2)$$

(*d*) The American Railway Association has adopted five standard sizes of axles, the requirements for which, based on the above formulas, are given in the following table:

TABLE I.

Classification of Axles	Size of Journal, in.	Diameter of Axle at Center, in.	Length of Axle, in.	Height of Drop, ft.	Number of Blows	Maximum Permanent Set, in.
A	$3\frac{3}{4}$ by 7	$4\frac{1}{4}$	$83\frac{1}{4}$	18	5	$8\frac{3}{4}$
		$4\frac{3}{8}$		19		$8\frac{3}{8}$
		$4\frac{1}{2}$		20		8
B	$4\frac{1}{4}$ by 8	$4\frac{3}{4}$	$84\frac{1}{4}$	$22\frac{1}{2}$	5	$7\frac{1}{2}$
		$4\frac{7}{8}$		$23\frac{1}{2}$		$7\frac{1}{4}$
		5		25		7
C	5 by 9	$5\frac{3}{8}$	$86\frac{1}{2}$	29	5	$6\frac{1}{4}$
		$5\frac{1}{2}$		30		6
		$5\frac{5}{8}$		$31\frac{1}{2}$		$5\frac{3}{4}$
D	$5\frac{1}{2}$ by 10	$5\frac{7}{8}$	$88\frac{1}{2}$	$34\frac{1}{2}$	5	$5\frac{1}{2}$
		6		36		$5\frac{1}{4}$
		$6\frac{1}{8}$		$37\frac{1}{2}$		5
E	6 by 11	$6\frac{7}{16}$	$90\frac{3}{4}$	$41\frac{1}{2}$	5	$4\frac{3}{4}$
		$6\frac{9}{16}$		43		$4\frac{1}{2}$
		$6\frac{11}{16}$		$44\frac{1}{2}$		$4\frac{1}{4}$

(*e*) The maximum permanent set is the difference between the distance from a straight edge to the middle point of the axle, measured before the first blow, and the distance measured in the same manner after the blow. The straight edge shall rest only on the end collars or the ends of the axle.

(*f*) The temperature of the test axle shall be between 40 and 120° F. (4 and 50° C.).

Drop Test Machine. 7. The anvil of the drop-test machine shall be supported on 12 springs, as shown on the A.R.A. drawings, and shall be free to move in a vertical direction, and shall weigh 17,500 lb. The radii of the striking face of the tup and of the supports shall be 5 in.

Number of Tests. 8. One drop test shall be made from each melt. Not less than 15 tons shall be offered from any one melt unless otherwise specified.

IV. WORKMANSHIP AND FINISH

9. (*a*) All axles shall be made and finished in a workmanlike manner and shall conform to the sizes and shaped specified by the customer and all journals and wheel seats shall be rough turned. In centering, unless otherwise specified, 60-deg. centers shall be used, with large diameter of counter-sink not less than $\frac{7}{8}$ in. and with clearance drilled $\frac{1}{2}$ in. deep.

(*b*) Unless otherwise specified, axles shall be smooth forged, except the wheel seats and journals, which shall be rough turned.

Finish. 10. The axles shall be free from injurious defects and shall have a workmanlike finish.

V. PERMISSIBLE VARIATIONS AND WEIGHTS

Permissible Variations. 11. (*a*) The length of the axles shall not be less than that specified and not more than $\frac{1}{8}$ in. over.

(*b*) *Smooth Forged Axles.*—The diameters of the straight center portion of the axles and the black collars shall not be less than that specified, and shall not be more than $\frac{1}{4}$ in. over. The intermediate portion of the axles shall not be under the dimensions specified, and shall be forged to a straight taper in a workmanlike manner.

(*c*) *Rough Turned Axles.*—The diameters of the straight center portion and the black collars shall not be less than that specified. The straight center portion shall not be more than $\frac{1}{8}$ in. over, and the black collars not more than $\frac{1}{4}$ in. over the diameter specified. The intermediate portion of the axles shall not be under the dimensions specified, and shall be turned to a straight taper in a workmanlike manner.

(*d*) The rough-turned diameter of the journals and wheel seats shall be from $\frac{1}{8}$ to $\frac{1}{4}$ in. over the finished sizes to provide for proper finishing.

Weights. 12. (*a*) The maximum weights of smooth-forged and rough-turned axles given in Section 6 (*d*) in any shipment shall not exceed those given in Table II.

TABLE II.

Classification of Axles	Size of Journal, in.	Maximum Weight, lb.	
		Smooth Forged with Rough Turned Journals and Wheel Seats	Rough Turned All Over
A	$3\frac{3}{4}$ by 7	435	425
B	$4\frac{1}{4}$ by 8	530	520
C	5 by 9	705	695
D	$5\frac{1}{2}$ by 10	835	825
E	6 by 11	1015	1005

(*b*) Axles shown in Table II shall be billed at actual weight, except that all weight in excess of the above maximum figures shall be at the expense of the manufacturer.

VI. MARKING

Marking. 13. Each axle shall have the manufacturer's name or brand legibly stamped on one of the black collars, and the melt number, the month and year when made stamped on the other, unless otherwise specified.

VII. INSPECTION AND REJECTION

Inspection. 14. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the axles ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the axles are being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment.

(*b*) The purchaser may make the chemical tests to govern the acceptance or rejection of the axles in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

(*c*) All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

15. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 14 (*b*) shall be reported within five working days from the receipt of samples. **Rejection.**

(*b*) Axles which show injurious defects while being finished by the purchaser will be rejected, and the manufacturer shall be notified.

16. Samples tested in accordance with Section 14 (*b*), which represent rejected axles, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time. **Rehearing.**

TENTATIVE SPECIFICATIONS

FOR

STRUCTURAL SILICON STEEL[1]

Serial Designation: A 94 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1925.

Material Covered. 1. These specifications cover a special high-strength structural steel intended primarily for use as main stress-carrying material of structural members.

I. MANUFACTURE

Process. 2. The steel shall be made by the open hearth-process.

Discard. 3. A sufficient discard shall be made from each ingot to secure freedom from injurious piping and undue segregation.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 4. The steel shall conform to the following requirements as to chemical composition:

	Ladle Analysis	Check Analysis
Carbon, per cent	not over 0.40	not over 0.44
Phosphorus, per cent, Acid	" " 0.06	" " 0.075
Phosphorus, per cent, Basic	" " 0.04	" " 0.05
Sulfur, per cent	" " 0.05	" " 0.063
Silicon, per cent	" under 0.20	" under 0.18

Ladle Analyses 5. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of carbon, manganese, phosphorus, sulfur and silicon. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative and shall conform to the requirements specified in Section 4 for ladle analysis.

Check Analyses. 6. Analyses may be made by the purchaser from finished material representing each melt. The chemical composition thus determined shall conform to the requirements specified in Section 4 for check analysis.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests.

7. (*a*) The material shall conform to the following requirements as to tensile properties:

Tensile strength, lb. per sq. in.	80 000 – 95 000
Yield point, min., lb. per sq. in.	45 000
Elongation in 8 in., min., per cent	$\frac{1\,500\,000^{a}}{\text{tens. str.}}$
Reduction of area, min., per cent	30 [b]

[a] See Section 8.
[b] See Section 9.

(*b*) The yield point shall be determined by the drop of the beam of the testing machine when operated at a cross-head speed not greater than $\frac{1}{2}$ in. per minute. The testing machine shall not be stopped to obtain the drop of the beam.

Modifications in Elongation.

8. For material over $\frac{3}{4}$ in. in thickness, a deduction from the percentage of elongation specified in Section 7 (*a*) of 0.25 per cent shall be made for each increase of $\frac{1}{16}$ in. of the specified thickness above $\frac{3}{4}$ in., to a minimum of 14 per cent.

Modifications in Reduction of Area.

9. For material over $\frac{3}{4}$ in. in thickness, a deduction from the percentage of reduction of area specified in Section 7 (*a*) of 0.50 per cent shall be made for each increase of $\frac{1}{16}$ in. of the specified thickness above $\frac{3}{4}$ in., to a minimum of 24 per cent.

Bend Tests.

10. The test specimen shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion around a pin, the diameter of which shall have the following relation to the thickness of the specimen:

Specimen Thickness	Pin Diameter
Up to $\frac{3}{4}$ in., inclusive	1 times the thickness
Over $\frac{3}{4}$ in. to 1 in., inclusive	$1\frac{1}{2}$ " " "
Over 1 in. to $1\frac{1}{4}$ in., inclusive	2 " " "
Over $1\frac{1}{4}$ in.	$2\frac{1}{2}$ " " "

Test Specimens.

11. (*a*) Test specimens shall be prepared for testing from the material in its rolled or forged condition, except as specified in Paragraph (*b*).

(*b*) Test specimens for annealed material shall be prepared from the material as annealed for use, or from a short length of a full section similarly treated.

(*c*) Test specimens shall be taken longitudinally, and, except as specified in Paragraphs (*e*) and (*f*), shall be of the full thickness or section of material as rolled.

(*d*) Test specimens for plates, shapes and flats may be machined to the form and dimensions shown in Fig. 1, or with both edges parallel.

(*e*) Tension test specimens for material over 1 in. in thickness or diameter may be machined to a thickness or diameter of at least $\frac{3}{4}$ in., for a length of at least 9 in.

(*f*) Bend test specimens for material over $1\frac{1}{4}$ in. in thickness or diameter may be machined to a thickness or diameter of at least $\frac{3}{4}$ in. or to 1 by $\frac{1}{2}$ in. in section.

(*g*) The machined sides of rectangular bend test specimens may have the corners rounded to a radius not over $\frac{1}{16}$ in.

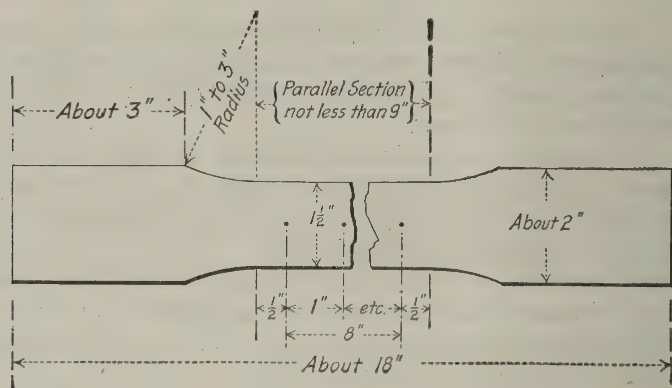

FIG. 1.

Number of Tests. 12. (*a*) Two tension and one bend test shall be made from each melt; except that if material from one melt differs $\frac{3}{8}$ in. or more in thickness, one tension and one bend test shall be made from both the thickest and the thinnest material rolled.

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 7 (*a*) and any part of the fracture is outside the middle third of the gage length, as indicated by marks placed on the specimen before testing, a retest shall be allowed.

IV. PERMISSIBLE VARIATIONS IN WEIGHT AND THICKNESS

Permissible Variations. 13. The cross-section or weight of each piece of steel shall not vary more than 2.5 per cent from that specified; except in the case of sheared plates, which shall be covered by the following permissible variations. One cubic inch of rolled steel is assumed to weigh 0.2833 lb.

(*a*) *When Ordered to Weight per Square Foot:* The weight of each lot[1] in each shipment shall not vary from the weight ordered more than the amount given in Table I.

TABLE I.—PERMISSIBLE VARIATIONS OF RECTANGULAR PLATES ORDERED TO WEIGHT.

Ordered Weight, lb. per sq. ft.	Permissible Variations in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Ordered Weights																		Ordered Weight, lb. per sq. ft.
	Under 48 in.		48 to 60 in., excl.		60 to 72 in., excl.		72 to 84 in., excl.		84 to 96 in., excl.		96 to 108 in., excl.		108 to 120 in., excl.		120 to 132 in., excl.		132 in. or over		
	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	
Under 5........	5	3	5.5	3	6	3	7	3	...	...	...	...	...	...	...	...	...	...	Under 5
5 to 7.5 excl.	4.5	3	5	3	5.5	3	6	3	...	...	...	...	...	...	...	...	...	...	5 to 7.5 excl.
7.5 " 10 "	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	...	...	...	...	7.5 " 10 "
10 " 12.5 "	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	9	3	10 " 12.5 "
12.5 " 15 "	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	12.5 " 15 "
15 " 17.5 "	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	15 " 17.5 "
17.5 " 20 "	2.5	2	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	17.5 " 20 "
20 " 25 "	2	2	2.5	2	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	20 " 25 "
25 " 30 "	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	4.5	3	5	3	25 " 30 "
30 " 40 "	2	2	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	4.5	3	30 " 40 "
40 or over......	2	2	2	2	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	40 or over

NOTE.—The weight per square foot of individual plates shall not vary from the ordered weight by more than $1\frac{1}{3}$ times the amount given in this table.

TABLE II.—PERMISSIBLE OVERWEIGHTS OF RECTANGULAR PLATES ORDERED TO THICKNESS.

Ordered Thickness, in.	Permissible Excess in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Nominal Weights									Ordered Thickness, in.
	Under 48 in.	48 to 60 in., excl.	60 to 72 in. excl.	72 to 84 in., excl.	84 to 96 in., excl.	96 to 108 in., excl.	108 to 120 in., excl.	120 to 132 in., excl.	132 in. or over	
Under $\frac{1}{8}$...........	9	10	12	14	..	..	..	..	..	Under $\frac{1}{8}$
$\frac{1}{8}$ to $\frac{3}{16}$ excl.....	8	9	10	12	..	..	..	..	..	$\frac{1}{8}$ to $\frac{3}{16}$ excl.
$\frac{3}{16}$ " $\frac{1}{4}$ "	7	8	9	10	12	..	..	..	..	$\frac{3}{16}$ " $\frac{1}{4}$ "
$\frac{1}{4}$ " $\frac{5}{16}$ "	6	7	8	9	10	12	14	16	19	$\frac{1}{4}$ " $\frac{5}{16}$ "
$\frac{5}{16}$ " $\frac{3}{8}$ "	5	6	7	8	9	10	12	14	17	$\frac{5}{16}$ " $\frac{3}{8}$ "
$\frac{3}{8}$ " $\frac{7}{16}$ "	4.5	5	6	7	8	9	10	12	15	$\frac{3}{8}$ " $\frac{7}{16}$ "
$\frac{7}{16}$ " $\frac{1}{2}$ "	4	4.5	5	6	7	8	9	10	13	$\frac{7}{16}$ " $\frac{1}{2}$ "
$\frac{1}{2}$ " $\frac{5}{8}$ "	3.5	4	4.5	5	6	7	8	9	11	$\frac{1}{2}$ " $\frac{5}{8}$ "
$\frac{5}{8}$ " $\frac{3}{4}$ "	3	3.5	4	4.5	5	6	7	8	9	$\frac{5}{8}$ " $\frac{3}{4}$ "
$\frac{3}{4}$ " 1 "	2.5	3	3.5	4	4.5	5	6	7	8	$\frac{3}{4}$ " 1 "
1 or over	2.5	2.5	3	3.5	4	4.5	5	6	7	1 or over

(*b*) *When Ordered to Thickness:* The thickness of each plate shall not vary more than 0.01 in. under that ordered.

The overweight of each lot[1] in each shipment shall not exceed the amount given in Table II.

[1] The term "lot" as applied to Table I means all of the plates of each group width and group weight; as applied to Table II, it means all of the plates of each group width and group thickness.

V. FINISH

Finish. 14. The finished material shall be free from injurious defects and shall have a workmanlike finish.

VI. MARKING

Marking. 15. The name or brand of the manufacturer and the melt number shall be legibly stamped or rolled on all finished material. The melt number shall be legibly marked, by stamping if practicable, on each test specimen.

VII. INSPECTION AND REJECTION

Inspection. 16. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 17. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 6 shall be reported within five working days from the receipt of samples.

(*b*) Material which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing. 18. Samples tested in accordance with Section 6, which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS

FOR

CARBON-STEEL CASTINGS FOR VALVES, FLANGES AND FITTINGS FOR HIGH-TEMPERATURE SERVICE[1]

Serial Designation: A 95 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1925.

Material Covered. 1. These specifications cover carbon-steel castings for valves, flanges and fittings for service up to 750° F. (400° C.) total temperature.

I. MANUFACTURE

Process. 2. The steel shall be made by one or more of the following processes: open-hearth, electric-furnace, converter, or crucible.

Heat Treatment. 3. (*a*) All castings shall receive a heat treatment proper to the design and chemical composition of the castings.

(*b*) Heat treatment, unless otherwise specified by the purchaser, may consist of annealing, or of normalizing, or of normalizing followed by annealing, or of normalizing followed by a draw-back to a temperature below the critical range. No castings which have been quenched in any liquid medium shall be offered under these specifications.

(*c*) *Annealing*.—The procedure for annealing shall consist in allowing the castings to cool, after pouring, to a temperature below the critical range. They shall then be uniformly reheated to the proper temperature to refine the grain and allowed to cool uniformly in the furnace.

(*d*) *Normalizing*.—The procedure for normalizing shall consist in allowing the castings to cool, after pouring, to a temperature below the critical range. They shall then be reheated to the proper temperature to refine the grain and allowed to cool in still air.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 4. (*a*) The castings shall conform to the following requirements as to chemical composition, except that the following limitations for

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

carbon, manganese and silicon are intended to show desired composition and are not to be used for rejection:

Carbon	0.15 – 0.40	per cent
Manganese	not under 0.50	"
Silicon	not under 0.20	"
Phosphorus	not over 0.05	"
Sulfur	not over 0.05	"

(*b*) Manufacturers submitting proposals to furnish material under these specifications shall specify, with their proposals, the limits of carbon, manganese and silicon which they will accept as a basis for rejection.

Ladle Analyses. 5. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of the elements specified in Section 4. This analysis shall be made from drillings taken at least $\frac{1}{4}$ in. beneath the surface of a test ingot obtained during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative and shall conform to the requirements specified in Section 4.

Check Analyses. 6. Analyses may be made by the purchaser from the broken tension test specimen or from a casting representing each melt. The chemical composition thus determined shall conform to the requirements specified in Section 4. Samples for analysis shall be taken not less than $\frac{1}{4}$ in. beneath the surface, except in the case of castings which are too thin, and shall be taken in such a manner as not to impair the usefulness of the casting.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests. 7. (*a*) The castings shall conform to the following minimum requirements as to tensile properties:

Tensile strength, lb. per sq. in.	70 000
Yield point, lb. per sq. in.	36 000
Elongation in 2 in., per cent	22
Reduction of area, per cent	30

(*b*) The yield point shall be determined by the drop of the beam of the testing machine at a cross-head speed not to exceed $\frac{1}{8}$ in. per minute. The tensile strength shall be determined at a speed not to exceed $1\frac{1}{2}$ in. per minute.

Bend Test. 8. (*a*) Bend tests shall be required for castings only when so specified in the order.

(*b*) When a bend test is specified, the test specimens shall stand being bent cold through an angle of 90 deg. around a pin 1 in. in diameter, without cracking on the outside of the bent portion.

9. (*a*) Tension test specimens, and, when a bend test is specified, bend test specimens, shall be taken from test bars cast attached to the castings where practicable. If the design of the castings is such that test bars should not be attached to the castings, the test bars shall be cast attached to special blocks, a sufficient number of which shall be provided for each lot of castings. Test bars from which tension and bend test specimens are to be taken shall remain attached to the castings or blocks they represent through heat treatment and until presented for inspection. Test bars shall be provided in sufficient numbers to furnish the tests required in Section 10. **Test Specimens.**

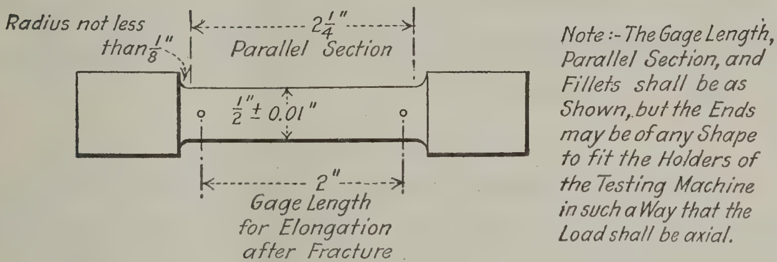

FIG. 1.

(*b*) If satisfactory to the manufacturer and inspector, tension test specimens may be cut from heat-treated castings, instead of from test bars.

(*c*) Tension test specimens shall conform to the dimensions shown in Fig. 1. The ends shall be of a form to fit the holders of the testing machine in such a way that the load shall be axial.

(*d*) Bend test specimens shall be machined to 1 by $\frac{1}{2}$ in. in section with the corners rounded to a radius of not over $\frac{1}{16}$ in.

10. (*a*) One tension test and, when specified, one bend test shall be made from each melt in each heat-treatment charge and, when specified, from each casting weighing 500 lb. or over. **Number of Tests.**

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded; in which case another specimen from the same lot shall be substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 7 (*a*) and any part of the fracture is more than $\frac{3}{4}$ in. from the center of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Retests. 11. If the results of the physical tests for any lot do not conform to the requirements specified, such lot may be re-treated, but not more than twice. Retests shall be made as specified in Sections 7 and 8.

IV. WORKMANSHIP AND FINISH

Workmanship. 12. The castings shall conform substantially to the shapes and sizes indicated by the patterns and drawings submitted by the purchaser.

Finish. 13. (*a*) The castings shall be free from injurious defects.

Welding. (*b*) Defects which do not impair the strength of the castings may be welded by an approved process. The defects shall be cleaned out to solid metal, before welding, and when so required by the inspector, shall be submitted to him in this condition for his approval. All castings shall be heat-treated after welding in accordance with the requirements in Section 3.

V. INSPECTION AND REJECTION

Inspection. 14. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the castings ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the castings are being furnished in accordance with these specifications.

(*b*) If, in the case of important castings for special purposes, surface inspection in the green state is required, this shall be so specified in the order.

(*c*) All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 15. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 6 shall be reported within five working days from the receipt of samples.

(*b*) Castings which show injurious defects subsequent to their acceptance at the manufacturer's works will be rejected and the manufacturer shall be notified.

Rehearing. 16. Samples tested in accordance with Section 6, which represent rejected castings, shall be preserved for two weeks from the date of transmission of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS

FOR

ALLOY-STEEL BOLTING MATERIAL FOR HIGH-TEMPERATURE SERVICE[1]

Serial Designation: A 96 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Material Covered.

1. (*a*) These specifications cover alloy-steel bolting material for valves, flanges and fittings for temperatures from 400 to 750° F. (205 to 400° C.), inclusive. The term "bolting material" as used in these specifications covers rolled, forged or cold-drawn bars, and bolts, screws and studs.

(*b*) Three classes of material are covered, A, B and C, classified in accordance with their physical properties as specified in Section 10.

(*c*) Nickel, chrome-nickel, chrome-vanadium, chrome-manganese or any other types of alloy steel approved by the purchaser may be submitted under these specifications.

Basis of Purchase.

2. When agreed upon between the manufacturer and the purchaser, Brinell hardness tests may be made to determine the acceptance of bolting material in lieu of the tension tests herein specified; but shall not be used as a basis for rejection without confirming tension tests being made.

Certification of Test for Bolts, Screws and Studs.

3. Unless otherwise specified on the order, a certification that bolts, screws and studs for valves and other fittings intended for stock and other purposes requiring assembly in the manufacturer's plant conform to the requirements of these specifications, shall be accepted in lieu of the tests herein specified.

I. MANUFACTURE

Process.

4. The steel shall be made by one or more of the following processes: open-hearth, electric-furnace or crucible.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

Discard. 5. A sufficient discard shall be made from each ingot to secure reedom from injurious piping and undue segregation.

Heat Treatment. 6. (*a*) Heat treatment shall consist of quenching and tempering.

(*b*) Immediately after rolling or forging, the bolting material shall be allowed to cool to a temperature below the critical range under suitable conditions to prevent injury by too rapid cooling. For quenching and tempering, the material shall then be uniformly reheated to the proper temperature to refine the grain (a group thus reheated being known as a "quenching charge") and quenched in some medium under substantially uniform conditions for each quenching charge. The material shall then be uniformly reheated to the proper temperature for tempering or "drawing back" (a group thus reheated being known as a "tempering charge") and allowed to cool uniformly.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 7. (*a*) The steel shall conform to the following requirements as to chemical composition:

Phosphorus, per cent	not over 0.04
Sulfur, per cent	not over 0.05

(*b*) The composition of the steel furnished under these specifications, other than phosphorus and sulfur, shall be agreed upon by the manufacturer and the purchaser.

NOTE.—It is recommended that the carbon content shall not be less than 0.20 nor more than 0.45 per cent, and that the carbon ranges shall be 0.10 per cent of carbon.

Ladle Analyses. 8. An analysis of each melt of steel shall be made by the steel manufacturer to determine the percentages of the elements present. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 7.

Check Analyses. 9. Analyses may be made by the purchaser from samples representing the bolting material. The chemical composition thus determined shall conform to the requirements specified in Section 7, except that the rejection limit for phosphorus shall be 0.05 per cent and for sulfur 0.055 per cent.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests. 10. (*a*) The bolting material, after final heat treatment, shall conform to the following requirements as to tensile properties:

	Class A	Class B	Class C
Tensile strength, lb. per sq. in..	95 000 – 115 000	105 000 – 125 000	not less than 125 000
Yield point, min., lb. per sq. in..	70 000	80 000	105 000
Elongation in 2 in., min., per cent	20	20	16
Reduction of area, min., per cent	50	50	50

(*b*) The yield point shall be determined by the drop of the beam of the testing machine or by the use of dividers.

Brinell Hardness Tests.

11. (*a*) When Brinell hardness tests are made, the bolting material, after final heat treatment, shall conform to the following requirements:

	Brinell Hardness Number
Class A	190 – 250
Class B	210 – 270
Class C	260 – 320

(*b*) The Brinell hardness test shall be made in accordance with the Tentative Methods of Brinell Hardness Testing of Metallic Materials (Serial Designation: E 10 – 25 T) of the American Society for Testing Materials.[1]

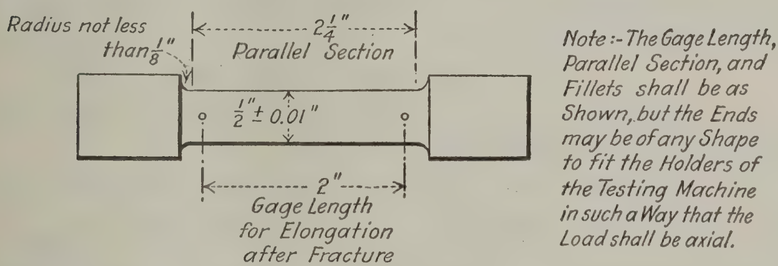

FIG. 1.

Tension Test Specimens.

12. (*a*) The tension test specimens taken from the bolting material shall conform to the dimensions shown in Fig. 1, except as specified in Paragraph (*b*). The ends shall be of a form to fit the holders of the testing machine in such a way that the load shall be axial.

(*b*) In the case of small sections which will not permit of taking the standard tension test specimen specified in Paragraph (*a*), the tension test specimen shall be as large as feasible and its dimensions shall be proportional to those shown in Fig. 1. The gage length for measuring elongation shall be four times the diameter of the specimen.[2]

[1] See p. 788.

[2] These requirements are in accordance with the provisions of Section 5 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 – 25 T), see p. 770.

Number of Tests. 13. (*a*) Except as specified in Paragraph (*b*), one tension test shall be made for each lot of 100 pieces or less of bolting material from each tempering charge. If more than one quenching charge is represented in a tempering charge, one tension test shall be made from each lot of 100 pieces or less from each quenching charge. If more than one melt is represented in a quenching charge, one tension test shall be made from each lot of 100 pieces or less from each melt.

(*b*) Unless required on the order, tension tests shall not be made on an order of less than 100 bolts, screws or studs; in which case acceptance shall be based upon the provisions in Sections 2 or 3.

(*c*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*d*) If the percentage of elongation of any test specimen is less than that specified in Section 10 (*a*) and any part of the fracture is outside of the middle three-fourths of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Retests. 14. If the results of the physical tests of any lot of bolting material do not conform to the requirements specified, the manufacturer may re-treat such lot one or more times and retests shall be made as specified in Section 13 (*a*).

IV. WORKMANSHIP AND FINISH

Finish. 15. The bolting material shall be free from injurious defects and shall have a workmanlike finish.

Nuts and Washers. 16. Bolts and studs shall be equipped with cold-punched semi-finished carbon-steel nuts of U. S. Standard rough dimensions, chamfered and trimmed. If washers are used under nuts they shall be of forged or rolled steel. All nuts and washers shall be free from injurious defects and shall have a workmanlike finish, and shall conform to the chemical requirements for phosphorus and sulfur specified in Sections 7 (*a*) and 9.

Threads. 17. U. S. Standard threads shall be used, except that no stud shall have less than eight threads per inch.

V. MARKING

Identification Marks. 18. Identification marks to be stamped on bolting material shall be agreed upon between the manufacturer and the purchaser.

VI. INSPECTION AND REJECTION

Inspection. 19. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is

being performed, to all parts of the manufacturer's works which concern the manufacture of the bolting material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the bolting material is being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment.

(*b*) The purchaser may make the tests to govern the acceptance or rejection of the bolting material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

(*c*) Tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection.

20. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 19 (*b*) shall be reported within five working days from the receipt of samples.

(*b*) Bolting material which shows injurious defects while being finished by the purchaser will be rejected and the manufacturer shall be notified.

Rehearing.

21. Samples tested in accordance with Section 19 (*b*), which represent rejected bolting material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the test, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
TUNGSTEN POWDER[1]

Serial Designation: A 97 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades. 1. These specifications cover tungsten powder in one grade only.

Basis of Purchase. 2. The tungsten powder shall be thoroughly mixed and packed in sound containers preferably metallic or metallic lined and sufficiently strong to prevent loss in transportation.

Chemical Requirements. 3. The material shall conform to the following requirements as to chemical composition:

Metallic Tungsten, minimum	95.00 per cent
Carbon, maximum	0.50 "
Sulfur, maximum	0.05 "
Phosphorus, maximum	0.05 "
Oxygen, maximum	1.00 "
Silicon, maximum	0.75 "
Tin, maximum	0.03 "
Copper, maximum	0.03 "
Arsenic, maximum	0.03 "
Bismuth, maximum	0.03 "
Antimony, maximum	0.03 "

Sampling. 4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 – 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis. 5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 – 25 T) of the American Society for Testing Materials.[3]

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 85. [3] See p. 90.

TENTATIVE SPECIFICATIONS

FOR

SPIEGELEISEN[1]

Serial Designation: A 98 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

1. These specifications cover two grades of spiegeleisen, as follows: **Grades.**

Grade A; and
Grade B.

2. The spiegeleisen shall be furnished in pigs or lumps and each shipment shall be uniform as to the grade specified. **Basis of Purchase.**

3. The material shall conform to the following requirements as to chemical composition: **Chemical Requirements.**

	Manganese, per cent	Carbon, max., per cent	Phosphorus, max., per cent	Sulfur, max., per cent	Silicon, max., per cent
Grade A......	19 to 21	6.50	0.15	0.04	as specified
Grade B......	16 to 19	6.50	0.25	0.05	as specified

4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 – 25 T) of the American Society for Testing Materials.[2] **Sampling.**

5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 – 25 T) of the American Society for Testing Materials.[3] **Methods of Chemical Analysis.**

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 85. [3] See p. 90.

TENTATIVE SPECIFICATIONS
FOR
FERRO-MANGANESE[1]

Serial Designation: A 99 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades. 1. These specifications cover ferro-manganese in one grade only.

Basis of Purchase. 2. Ferro-manganese shall be furnished in pigs, lumps, crushed or screened to size, as specified.

Chemical Requirements. 3. The material shall conform to the following requirements as to chemical composition:

Manganese, minimum	78.00 per cent
Phosphorus, maximum	0.35 "
Carbon, maximum	7.5 "
Silicon, maximum	1.00 "
Sulfur, maximum	0.050 "

Sampling. 4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 – 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis. 5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 – 25 T) of the American Society for Testing Materials.[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 85. [3] See p. 90.

TENTATIVE SPECIFICATIONS
FOR
FERRO-SILICON[1]

Serial Designation: A 100 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades.

1. These specifications cover three grades of ferro-silicon, as follows:

Grade A;
Grade B; and
Grade C.

Basis of Purchase.

2. (*a*) The ferro-silicon shall be furnished in lumps, or crushed or screened to size, as specified.

(*b*) When furnished in lump or granulated form the ferro-silicon shall be free from excessive disintegration.

Chemical Requirements.

3. The material shall conform to the following requirements as to chemical composition:

	GRADE A	GRADE B	GRADE C
Silicon, per cent...........	47.00 to 53.00	72.00 to 78.00	85.00 to 95.00

4. An analysis of each shipment of ferro-silicon shall be furnished the purchaser, showing the percentage of silicon.

Sampling.

5. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 – 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis.

6. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104–25 T) of the American Society for Testing Materials.[3]

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 85. [3] See p. 90.

TENTATIVE SPECIFICATIONS
FOR
FERRO-CHROMIUM[1]

Serial Designation: A 101 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades.

1. These specifications cover ferro-chromium in four grades, as follows:

High Carbon, Grade A;
Low Carbon, Grades B, C and D.

Basis of Purchase.

2. *Ferro-Chromium, Low Carbon.*—This material shall be crushed to the specified size and mixed before packing, so that the quality in each package is uniform with the lot.

Ferro-Chromium, High-Carbon.—This material shall be furnished to size as specified.

Chemical Requirements.

3. The material shall conform to the following requirements as to chemical composition:

	High Carbon	Low Carbon		
	Grade A	Grade B	Grade C	Grade D
Chromium, per cent	60.00 to 75.00	60.00 to 75.00	60.00 to 75.00	60.00 to 75.00
Carbon, per cent	4.00 to 8.00 as specified	1.50 to 2.00	1.00 to 1.50	under 1.00 as specified[a]
Silicon, per cent	as specified	as specified	as specified	as specified

[a] Grade D material may be obtained with various maxima carbon contents down to 0.10 per cent of carbon.

Sampling.

4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 – 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis.

5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 – 25 T) of the American Society for Testing Materials.[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 85. [3] See p. 90.

TENTATIVE SPECIFICATIONS
FOR
FERRO-VANADIUM[1]

Serial Designation: A 102 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Grades.

1. These specifications cover ferro-vanadium in four grades, as follows:

Grade A;
Grade B;
Grade C; and
Grade D.

Basis of Purchase.

2. The material furnished under this specification shall be crushed to the specified size, and mixed before packing, so that the quality in each package is uniform with the lot. The material shall be packed in sound containers, sufficiently strong to prevent loss in transportation.

Chemical Requirements.

3. The material shall conform to the following requirements as to chemical composition:

	Grade A	Grade B	Grade C	Grade D
Vanadium, per cent..........	30.00 to 40.00	30.00 to 40.00	35.00 to 45.00	35.00 to 45.00
Carbon, per cent.............	3.00 to 6.00	1.5 to 3.00	not over 1.5	not over 0.75
Silicon, per cent.............	8.00 to 15.00	5.00 to 8.00	not over 2.00	not over 2.00
Phosphorus, per cent..........	not over 0.250	not over 0.250	not over 0.150	not over 0.100
Sulfur, per cent..............	not over 0.300	not over 0.300	not over 0.200	not over 0.100
Aluminum, per cent...........	not over 2.00	not over 2.00	not over 2.00	not over 1.00

Sampling.

4. When agreed upon by the purchaser and seller the sampling shall be carried out in accordance with the Tentative Methods of Sampling Ferro-Alloys (Serial Designation: A 103 – 25 T) of the American Society for Testing Materials.[2]

Methods of Chemical Analysis.

5. When agreed upon by the purchaser and seller the chemical analysis shall be carried out in accordance with the Tentative Methods of Chemical Analysis of Ferro-Alloys (Serial Designation: A 104 – 25 T) of the American Society for Testing Materials.[3]

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 85. [3] See p. 90.

TENTATIVE SPECIFICATIONS

FOR

HOLLOW STAYBOLT IRON.[1]

Serial Designation: A 86 – 25 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1925.

I. MANUFACTURE.

Process.

1. The iron shall be rolled from a bloom, slab pile or box pile, made wholly from reworked puddled pig iron or reworked knobbled charcoal iron. The puddle mixture and the component parts of the bloom, slab pile or box pile shall be free from any admixture of iron scrap or steel.

Definition of Terms.

2. (*a*) *Bloom.*—A bloom is a solid mass of iron that has been hammered into a convenient size for rolling.

(*b*) *Slab pile.*—A slab pile is built up wholly of flat bars of iron of the full length of the pile.

(*c*) *Box pile.*—A box pile is a pile, the sides, top and bottom of which are formed by four flat bars and the interior of which consists of a number of small bars the full length of the pile.

(*d*) *Iron Scrap.*—This term applies only to foreign or bought scrap and does not include local mill products free from foreign or bought scrap.

II. CHEMICAL PROPERTIES AND TESTS.

Chemical Composition.

3. The iron shall conform to the following requirement as to chemical composition:

Manganese...not over 0.10 per cent.

Check Analyses.

4. (*a*) An anlysis may be made by the purchaser from a broken tension test specimen representing each lot as specified in Section 10 (*a*). The chemical composition thus determined shall conform to the requirement specified in Section 3.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. C. C. Osterhout, Secretary of Committee A-2 on Wrought Iron, Rome Iron Mills, Inc., Rome, N. Y.

(*b*) Drillings for analysis shall be so taken as to represent the entire cross-section of the specimen.

III. PHYSICAL PROPERTIES AND TESTS.

Tension Tests.

5. (*a*) The iron shall conform to the following requirements as to tensile properties:

Tensile strength, lb. per sq. in.	48 000 – 52 000
Yield point, min., lb. per sq. in.	0.6 tens. str.
Elongation, when measured in 8 in., min., per cent	28
Elongation, " " " 4 " " "	35
Reduction of area, min., per cent	42

(*b*) The yield point shall be determined by the drop of the beam of the testing machine. The speed of the cross-head of the machine shall not exceed $\frac{3}{4}$ in. per minute.

(*c*) In calculating the tensile strength and elastic limit, the area of a $\frac{7}{32}$-in. hole shall be deducted from the area of the bar, but the area of the hole shall be disregarded in calculating the reduction of area.

Bend Tests.

6. (*a*) The test specimen shall withstand being bent cold through 180 deg. flat on itself in both directions, without fracture on the outside of the bent portion.

(*b*) Bend tests may be made by pressure or by blows.

Splitting Tests.

7. A piece not less than 2 in. long shall be split open from end to end by driving a drift or wedge through the hole. The structure thus exhibited shall be free from signs of imperfect welding and the presence of slag or scale in the welds.

Etch Tests.

8. The cross-section of the test specimen shall be ground or polished, and etched[1] for a sufficient period to develop the structure. This test shall show the material to have been rolled from a bloom, slab pile or box pile, and to be free from steel.

Test Specimens.

9. All test specimens shall be of the full section of material as rolled.

Number of Tests.

10. (*a*) Bars of one size shall be sorted into lots of 1600 lineal feet. Two samples shall be selected at random from each lot and tested as specified in Sections 5, 6 and 7; but only one of these shall be tested as specified in Sections 4 and 8.

(*b*) If any of the samples originally selected to represent a lot of material contains surface defects not visible before testing but visible after testing, or if a tension test specimen breaks outside the middle third of the gage length, the individual sample shall be rejected and one retest from a different bar or bolt will be allowed.

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[1] A solution of 10 per cent hydrochloric acid (sp. gr. 1.19), 30 per cent sulfuric acid (sp. gr. 1.84), and 60 per cent water; or 25 per cent nitric acid (sp. gr. 1.42), and 75 per cent water, is recommended for the etch test.

IV. PERMISSIBLE VARIATIONS IN GAGE.

Permissible Variations. 11. The bars shall be truly round within 0.01 in. and shall be not less than 0.005 in. over, nor more than 0.020 in. over the specified diameter.

V. FINISH.

Finish. 12. The bars shall be smoothly rolled and free from slivers, depressions, seams, crop ends, and evidences of being burnt.

VI. MARKING.

Marking. 13. The material shall be suitably marked for identification.

VII. INSPECTION AND REJECTION.

Inspection. 14. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, free of charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment.

(*b*) The purchaser may make the tests to govern the acceptance or rejection of material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

(*c*) All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 15. (*a*) If either of the samples selected to represent a lot does not conform to the requirements specified in Sections 3, 5, 6, 7, 8 and 9, the lot will be rejected.

(*b*) Unless otherwise specified, any rejection based on tests made in accordance with Section 14 (*b*) shall be reported within five working days from the receipt of samples.

(*c*) Bars which will not take a clean, sharp thread with dies in fair condition, or which develop defects in forging or machining, will be rejected, and the manufacturer shall be notified.

Rehearing. 16. Samples tested in accordance with Section 14 (*b*), which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS

FOR

HOT-DIPPED GALVANIZED SHEETS[1]

Serial Designation: A 93 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

1. These specifications cover hot-dipped bessemer steel, open-hearth steel, open-hearth iron and wrought-iron sheets of three classes, as follows: **Scope.**

Class A—Flat or corrugated sheets that are not intended to be formed.

Class B—Flat or corrugated sheets to be curved to a large radius.

Class C—Flat sheets for miscellaneous purposes.

I. CHEMICAL PROPERTIES AND TESTS

2. These specifications contain no detailed requirements for the chemical composition of the base metal. If base metal containing copper is specified, it shall conform to the following requirements: **Chemical Composition.**

Copper, minimum.................................... 0.20 per cent

3. An analysis of copper-bearing sheets may be made by the purchaser from the test specimen specified in Section 6. The copper content thus determined shall conform to the requirement specified in Section 2. **Check Analysis.**

4. (*a*) The weight of the zinc coating shall conform to the requirements specified in Table I, the average being taken of the chemical determination from three specimens $2\frac{1}{4}$ in. square cut from the ends and middle of the sheet as specified in Section 7; provided, however, that the coating on any one of the three specimens shall be at least 75 per cent of the minimum average weight of coating specified in Table I. **Zinc Coating.**

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. James Aston, Secretary of Committee A-5 on Corrosion of Iron and Steel, A. M. Byers Co., Pittsburgh, Pa.

(*b*) The weight of the zinc coating shall be determined by the basic lead acetate, hydrochloric acid - antimony chloride or sulfuric-permanganate method.

(*c*) The weight of coating specified in Table I is the total coating on both sides of a sheet 1 ft. square, the numerical value of which in ounces is the same as that determined in grams from a sample $2\frac{1}{4}$ in. square.

II. PHYSICAL PROPERTIES AND TESTS

Bend Tests. 5. (*a*) *Base Metal Tests.*—Any portion of the test specimen specified in Section 6 shall bend cold through 180 deg. flat on itself without fracture of the base metal. For steel, the bend may be made in any direction; for wrought iron the line of the bend shall be transverse to the direction of the rolling of the sheet.

Table I.—Weight of Coating.

Galvanized Sheet Gage	Minimum Average Weight of Coating, oz. per sq. ft. Class A	Class B	Class C
No. 8	2.50	2.00	1.75
No. 9	2.50	2.00	1.75
No. 10	2.50	2.00	1.75
No. 11	2.50	2.00	1.75
No. 12	2.50	2.00	1.75
No. 14	2.50	2.00	1.75
No. 16	2.50	2.00	1.75
No. 18	2.50	2.00	1.50
No. 20	2.50	2.00	1.50
No. 22	2.50	2.00	1.50
No. 24	....	2.00	1.30
No. 26	....	2.00	1.10
No. 27	....	2.00	1.10
No. 28	....	1.80	1.10
No. 29	....	1.60	1.10
No. 30	....	1.40	1.10

(*b*) *Coating Tests.*—Sheets of Class *A* material shall stand corrugating without flaking or cracking of the coating. Sheets of Class *B* material shall stand corrugating and also subsequent longitudinal curving, as given below, without flaking or cracking of the coating.

Gage	Diameter of Circle
No. 12	36 in.
No. 14	21 "
No. 16	12 "

In the case of sheets of Class *C* material, any portion of the test specimen specified in Section 6, of sheets lighter than No. 16 gage,

shall stand bending in any direction without flaking of the coating on either side when bent through 180 deg. over four thicknesses of the material. A vise with smooth jaws and operated by hand shall be used for making the bend tests.

Test Specimens. 6. Three specimens exactly $2\frac{1}{4}$ in. square shall be cut to represent each test sheet, one being cut from the center of the sheet and the others from diagonally opposite corners, but at least 4 in. from the end and 2 in. from the side of the sheet.

Number of Tests. 7. (*a*) One each of the tests specified shall be made from one sheet in each lot of 1000 sheets or fraction thereof of each gage of each lot offered for inspection.

(*b*) If any sheet fails to meet the requirements of these specifications, a retest will be allowed from two other sheets in the same lot, both of which shall meet the requirements.

III. PERMISSIBLE VARIATIONS

Dimensions. 8. The sheets shall conform to the dimensions specified with the following permissible variations:

(*a*) The width shall be not less than that ordered, but may be $\frac{1}{4}$ in. over for sheets 48 in. or less in width, and $\frac{3}{8}$ in. over for sheets over 48 in. in width.

(*b*) The length shall be not less than that ordered, but for sheets 96 in. or less in length it may be $\frac{3}{4}$ in. greater. For each 24 in. or fraction thereof above 96 in., the permissible variation may be increased $\frac{1}{4}$ in. over $\frac{3}{4}$ in.

(*c*) When sheets are required accurate to size, they shall be ordered "to be re-squared."

Weight. 9. The average weight per square foot of the galvanized sheets as determined by the weighing of lots not exceeding 6000 lb. shall conform to the requirements specified in Table II, which are based on a maximum variation from the nominal weight of 5 per cent for gages Nos. 8 to 16, inclusive, and 3.5 per cent for gages thinner than Nos. 16 to 22, inclusive, and 2.5 per cent for gages thinner than No. 22.

IV. FINISH

Finish. 10. The finished sheets shall be of first-class commercial quality, free from flaws or mechanical defects such as pin holes, cracks, blisters, and blackened sal-ammoniac or acid spots.

V. MARKING

Marking. 11. The top sheet in each bundle, the top sheet in each lot or a number of sheets when shipped loose shall legibly show the name and brand of the manufacturer, class, gage and size.

VI. INSPECTION AND REJECTION.

Inspection. 12. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications.

(*b*) The purchaser may make the tests and inspection to govern the acceptance or rejection of the material in his own laboratory or elsewhere. Such tests and inspection shall be made at the expense of the purchaser.

Table II.—Weights of Galvanized Sheets.

Galvanized Sheet Gage	Weight of Galvanized Sheets, All Classes, oz. per sq. ft.		
	Minimum	Nominal	Maximum
No. 8	106.9	112.5	118.1
No. 9	97.4	102.5	107.6
No. 10	87.9	92.5	97.1
No. 11	78.4	82.5	86.6
No. 12	68.9	72.5	76.1
No. 14	49.9	52.5	55.1
No. 16	40.4	42.5	44.6
No. 18	33.3	34.5	35.7
No. 20	25.6	26.5	27.4
No. 22	21.7	22.5	23.3
No. 24	18.0	18.5	19.0
No. 26	14.1	14.5	14.9
No. 27	13.2	13.5	13.8
No. 28	12.2	12.5	12.8
No. 29	11.2	11.5	11.8
No. 30	10.2	10.5	10.8

Rejection. 13. (*a*) Sheets represented by samples which fail to conform to the requirements of these specifications will be rejected.

(*b*) Sheets which, subsequent to tests and inspection at the mill or elsewhere and their acceptance, show blackened sal-ammoniac or acid spots, improper galvanizing, improper trimming or other defects for which the manufacturer is responsible, will be rejected and shall be replaced by the manufacturer.

Rehearing. 14. Samples tested in accordance with Section 7 (*a*) which represent rejected material, shall be preserved for two weeks from the date of the test report.

TENTATIVE METHODS
OF
SAMPLING FERRO-ALLOYS[1]

Serial Designation: A 103 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

The procedures recommended in these methods are considerably more elaborate than those in general use by the principal consumers of ferro-alloys in this country. However, the methods have been designed as standard procedure for use in important cases where large sums are often involved and they accordingly should represent the most approved modern practice.

FERRO-SILICON, HIGH-CARBON FERRO-MANGANESE AND SILICO-MANGANESE

These alloys are usually shipped in bulk in carloads and should be sampled while being loaded or unloaded. At these times the sampler can make the fairest estimate of the ratio of lump to fine material and should secure proportionate amounts of each. Frequently the percentage of fines is negligible.

While the shipment is being loaded or unloaded, samples aggregating 150 lb. or more shall be gathered from all parts of the car with due regard to the estimated ratio of lump to fine material. In breaking lumps or pigs, fragments representing the full cross-section should be secured, a heavy hammer or sledge being employed.

The gross sample thus obtained shall be crushed to pea size in a heavy crusher provided with smooth plates of manganese steel. The crushed product shall be repeatedly mixed and halved with a Jones divider until about 5 lb. remain. If a Jones divider is not available, the sample may be reduced by "quartering," but use of the divider is preferred. The sample shall be further reduced to approximately a size to pass a No. 10 sieve in laboratory rolls and again cut down by a Jones divider or by "quartering" to about 300 g. This sample shall then be divided in the same way into four portions of approximately 75 g. each and three of these shall be reserved. The fourth portion shall be pulverized until all has been passed through a No. 80

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

or No. 100 sieve. A steel mortar with a close-fitting pestle, both of hardened alloy steel, shall be used for this reduction. Rejection of any part of the sample because of failure to pass the screen is almost sure to produce error. The sample shall be thoroughly mixed, dried for one hour at 105 to 110° C. It is then ready for analysis. If necessary, one-half rather than one-fourth of the 10-mesh sample shall be pulverized to pass a No. 80 or No. 100 sieve and the resulting sample divided for distribution to the interested parties.

HIGH-CARBON FERRO-CHROMIUM, CHROMIUM METAL, LOW-CARBON FERRO-MANGANESE, MANGANESE METAL, FERRO-TUNGSTEN, FERRO-MOLYBDENUM AND FERRO-VANADIUM

These alloys are usually shipped in stout kegs or barrels. Most of these products are relatively costly, and accuracy in sampling is extremely important even when the tonnage involved is small.

If the shipment is believed to be fairly uniform in composition and contains only a small percentage of fines, a satisfactory sample can be obtained by removing the ends or cutting the sides of the packages and taking from each at least 0.5 per cent of the contents. Thus at least $2\frac{1}{2}$ lb. would be taken from a package containing 500 lb. If the product has been crushed to a small size, for example $\frac{1}{4}$ to $\frac{3}{4}$ in., the sample can be taken with a shovel or scoop. If the material is in larger pieces, as is usual with high-carbon ferro-chromium, the sample shall be taken by breaking fragments from a number of lumps until the requisite weight has been obtained as already described for the sampling of lumps of ferro-silicon. The gross sample shall be obtained by combining the samples taken from the individual packages and crushed and worked down in the manner described for the preceding group of alloys.

The relatively simple procedure of the preceding paragraph is justified only when experience has shown that the product being sampled contains no more than a small percentage of "fines" and is relatively uniform in composition. The "fines" usually differ somewhat in composition from the lumps and always work toward the bottom of the container while in transit. Products like tungsten powder or pulverized ferro-silicon (80 to 85 per cent Si) are also liable to segregate while in transit and should be sampled in a manner to avoid error due to the accumulation of a heavy constituent in the bottom of the container. The term "relatively uniform in composition" applies to a shipment or a sub-lot of a shipment in which the composition of any package is approximately the same as that of any other. Frequently a shipment comprises two or more lots, each

uniform in composition within itself, but differing in this respect from the other lots. In these cases, each lot shall be sampled separately. If the product is rather variable in composition or if it contains a considerable proportion of fines, all of the packages comprising the shipment or lot shall be emptied and from each shall be taken a sample amounting to at least 0.5 per cent of the contents. The percentage of fine and lump alloy in each barrel shall be estimated and the sampling conducted accordingly. In sampling a small lot which varies widely in composition within itself, it is good practice to empty all of the packages, combine the entire contents and mix the lot thoroughly. The pile shall then be "quartered" repeatedly until a sample is obtained approximating 5 per cent of the weight of the entire lot. The gross sample obtained by combining samples from the individual barrels or by "quartering" the entire shipment shall be crushed and worked down in the manner described for the group containing ferro-silicon.

LOW-CARBON FERRO-CHROMIUM

Low-carbon ferro-chromium is ordinarily shipped in stout kegs. The alloy usually contains about 70 per cent of chromium together with maximum contents of carbon ranging from 0.1 to 2 per cent. The combination of hardness and toughness characteristic of this product, particularly of the lower carbon grades, makes it the most difficult by far of any of the ferro-alloys to sample properly. In view of the great importance of the accurate determination of the carbon content—especially of the alloy containing a maximum of 0.1 per cent of carbon—the utmost care must be taken to avoid contamination of the sample with fragments of steel from the tools. Fortunately low-carbon ferro-chromium is usually uniform in composition and free from "fines."

If the alloy is known to be reasonably uniform in composition, 10 lumps shall be drawn from each package in the shipment or sub-lot. This can be accomplished by removing the head or cutting the side of the keg. If the composition is variable, each package of the shipment or sub-lot shall be emptied and about 20 lumps taken at random. The number of lumps should be varied according to the net weight of the package and the average size of the lumps found in it. One fragment about $\frac{1}{8}$ in. in diameter shall be broken from each lump of the gross sample, using a heavy hammer and a plate of hardened alloy steel. As nearly as possible each fragment should offer a cross-section of the lump from which it was taken. The resulting sample shall be crushed to approximately a size to pass a No. 10 sieve by means of a mortar and pestle of hardened alloy steel. This material

shall be cut down with a Jones divider or "quartered" until 30 or 40 g. remain. This quantity shall then be pulverized to pass a No. 40 sieve by means of the mortar and pestle. Especially in pulverizing the grades relatively low in carbon, an air hammer is almost indispensable. The material passing a No. 40 sieve shall be split into two or four parts according to the requirements, by means of a Jones divider or by "quartering." The following procedure is recommended for those cases in which prolonged crushing has resulted in a powder which passes entirely through a No. 20 sieve but which passes a No. 40 sieve in part only. The entire sample shall be passed through a No. 20 sieve and then divided into fractions finer and coarser than No. 40. The fractions shall be weighed and preserved in separate packages upon which are recorded the percentages by weight of the two sizes. The fractions may be analyzed separately or a composite sample may be prepared for the determination of each element, by weighing and subsequently combining the proper proportions of the coarse and fine material.

REQUIREMENTS FOR MORTAR AND PESTLE

In the analysis of ferro-alloys, errors arising from contamination of the sample by iron from the sampling appliances are both serious and common. A rapid and accurate method for the determination of unalloyed metallic iron in ferro-chromium and in ferro-silicon is known, and by this means such contamination has been detected and measured. Contamination has often been due to iron from the mortar and pestle but can be avoided by the proper selection and treatment of the steel from which they are made. These tools are essential to the accurate sampling of ferro-alloys—especially low carbon ferro-chromium —and when made in accordance with the following requirements are suitable for this purpose.

Chromium-nickel steel containing approximately 1.5 per cent Cr, 3 per cent Ni, and 0.35 per cent C is recommended for both tools. For the mortar a cylinder 3 by 3 in. shall be drilled axially to a diameter of $1\frac{1}{2}$ in. and a depth of $2\frac{1}{2}$ in. The bottom of the hole shall be made perfectly flat and the entire inner surface brought to a smooth finish. The pestle shall be made from a rod originally $1\frac{1}{2}$ in. in diameter by 5 in. long, and shall be smoothly faced and turned to a final diameter slightly less than $1\frac{1}{2}$ in. The mortar and pestle shall be heated for several hours in a laboratory muffle furnace at 750 to 800° C. and hardened by quenching in oil. The upper part of the pestle shall be slightly softer than the remainder in order to decrease the tendency to shatter. Both tools shall be polished with abrasive paper for the removal of scale. Such a mortar and pestle, together with an air hammer will pulverize alloys which will resist other equipment.

Other approved methods for the preparation of the pulverized sample from the gross sample may be substituted for the above provided they do not lead to contamination or loss of any portion of it. For example, the gross sample may be crushed entirely by rolls or in the absence of power machinery may be broken by hand to the required size. In the entire absence of power equipment the gross sample should be sent to a point where suitable facilities are available.

TENTATIVE METHODS OF CHEMICAL ANALYSIS

OF

FERRO-ALLOYS[1]

Serial Designation: A 104 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

FERRO-SILICON

DETERMINATION OF SILICON

BY

FUSION WITH SODIUM PEROXIDE AND DEHYDRATION WITH HYDROCHLORIC ACID

Transfer 0.5000 g. (use 0.4000 g. for 90-per-cent silicon) of the finely powdered sample (passing a No. 150 sieve) to a 50-cc. pure iron crucible made from No. 20 gage (0.32 in. thickness) ingot iron, add approximately 13 g. of sodium peroxide. Mix *thoroughly* with a platinum or iron rod and carefully clean the rod of adhering particles by scraping with another rod. Cover the mixture with a layer of about 2 g. of additional peroxide. Heat the crucible and contents on a hot plate for 5 to 10 minutes to expel any water in the peroxide which would cause spattering in the subsequent fusion. Carefully fuse over a low flame by holding the crucible with a pair of tongs and slowly revolving it around the outer edge of the flame until the contents have melted down quietly. When the fusion is molten rotate the crucible carefully to stir up any unattacked particles on the bottom or sides, the crucible and contents being maintained at a low red heat. Just before completion of the fusion, which requires

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. Charles McKnight, Jr., Secretary of Committee A-9 on Ferro-Alloys, International Nickel Co., 67 Wall St., New York City.

only three or four minutes, increase the temperature to bright redness for a minute. If the reaction has proceeded too violently, due to too rapid heating, to the use of insufficient sodium peroxide, or to the lack of thorough mixing, appreciable loss will occur and the work should be repeated.

Cover the crucible and allow it to cool until the fusion has solidified, and tap the crucible, while still warm, several times on an iron plate, which will loosen the fused mass in a solid cake. When the crucible and cake have completely cooled, transfer the cake to a large (275-cc.) gold or preferably platinum dish. Cover the dish with a clock glass and cautiously add 50 cc. of cold water. When the reaction ceases, wash the small amount of adhering matter in the crucible into the dish with a little water. Cool the solution and carefully add an excess of hydrochloric acid. Evaporate to dryness, preferably on a steam bath, but do not bake at a temperature above 110° C. If platinum or gold dishes are not available for solution of the fused cake, the latter can be disintegrated with water in a pure nickel dish and the contents then transferred to a porcelain dish containing sufficient hydrochloric acid to provide an excess of acid. It is not desirable to dissolve the fusions directly in porcelain, because of the action of the alkaline solution.

When the residue is dry, allow the dish to cool. Add 10 cc. of hydrochloric acid (sp. gr. 1.13), cover the dish and heat gently for a few minutes. Dilute with 100 cc. of hot water, digest for a few minutes and filter through an 11-cm. paper. Wash about eight times with hot 5-per-cent hydrochloric acid, three times with warm 5-per-cent sulfuric acid and a few times with hot water. Reserve this paper until the second evaporation has been completed.

Evaporate the filtrate to dryness and bake the covered casserole or dish at 110° C. for one hour. Heating at a higher temperature is unnecessary and also disadvantageous. Cool, add 10 cc. of hydrochloric acid (1:1), digest on the steam bath for ten minutes, add 100 cc. of warm water, filter immediately and wash about eight times with cold 1-per-cent hydrochloric acid, twice with cold 5-per-cent sulfuric acid and a few times with cold water.

Place the paper and silica from the *second* dehydration in a large partially covered platinum crucible and char the paper without inflaming. Add the paper containing the silica from the first dehydration, char as before and then ignite carefully until the carbon has been burned off. Great care should be exercised in igniting the papers as the current of air produced by a burning filter is sufficient to carry finely divided silica out of the crucible. When the carbon

of the filter paper has been completely burned, gradually heat the tightly covered crucible to the full heat of a blast lamp and continue the heating at 1050 to 1100° C. for 25 minutes. Cool in a desiccator, weigh and check for constant weight by ten minutes' additional heating at the above temperature. Add sufficient sulfuric acid (1:1) to moisten the silica and then 15 to 20 cc. of pure hydrofluoric acid (48-per-cent). Evaporate to dryness, ignite and weigh. The loss represents silica which contains 46.72 per cent of silicon. A blank, not omitting the same amount of hydrofluoric acid, should be carried through all stages of the operation.

NOTE

The silica remaining in the filtrate after the second dehydration is practically negligible, averaging about 0.0006 to 0.0010 g. in hydrochloric dehydration and 0.0010 to 0.0015 g. in sulfuric dehydration.

FERRO-MANGANESE

DETERMINATION OF MANGANESE BY THE MODIFIED BISMUTHATE METHOD[1]

REAGENTS REQUIRED

Dilute Nitric Acid (sp. gr. 1.135).—Mix 300 cc. of nitric acid (sp. gr. 1.42) and 700 cc. of distilled water. Test for proper specific gravity.

Dilute Nitric Acid (3 : 97).—Mix 30 cc. of recently boiled nitric acid (sp. gr. 1.42) and 970 cc. of distilled water. Add a little sodium bismuthate and shake well.

Asbestos.—Asbestos for this work should be digested in hot nitric acid (sp. gr. 1.42) and then washed free from acid with distilled water.

Sodium Bismuthate.—This reagent should be of 80-per-cent grade and its oxidizing power tested as follows: Shake 0.5 g. of the reagent with 4 g. of potassium iodide and a little water in a stoppered flask. Add 15 cc. of hydrochloric acid and allow to stand in the dark, with occasional shaking until the bismuthate has entirely decomposed. Dilute to 300 cc. and titrate with 0.1 N sodium thiosulfate using starch as an indicator at the end. One cubic centimeter of 0.1 N $Na_2S_2O_3$ equals 0.0140 g. of $NaBiO_3$.

[1] *Journal of Industrial and Engineering Chemistry*, Vol. 16, p. 58 (1924).

Standard Potassium Permanganate Solution.—See the Determination of Chromium in Ferro-Chromium.[1] One cubic centimeter of 0.1 *N* $KMnO_4$ equals 0.001099 g. of manganese.

METHOD

(*A*) *Solution of the Sample*

All samples should be crushed to approximately a size to pass a No. 100 sieve and dried at 105 to 110° C.

With high-carbon 80-per-cent ferro-manganese, dissolve 0.25 g. of the prepared sample in 60 cc. of nitric acid (sp. gr. 1.42) in a 750-cc. Erlenmeyer flask, provided with a clock-glass cover or trap to prevent loss by spraying.

Add 2 g. of potassium nitrate and boil gently until the volume of the solution has been reduced to about 15 cc. Cool, add 10 cc. of H_2SO_4 (1:1) and evaporate to strong fumes of sulfuric acid. Cool somewhat, dilute the solution with 200 cc. of HNO_3 (sp. gr. 1.135), heat to boiling, remove from the heat and add sodium bismuthate a little at a time until a precipitate of manganese dioxide has formed. When this occurs, add sulfurous acid dropwise until the precipitate has dissolved and an excess of about 1 cc. has been provided to reduce any chromium to the trivalent form. Boil the solution about five minutes to completely expel oxides of nitrogen, dilute to 250 cc. with water, cool the flask and solution in crushed ice and treat as described later. High-carbon ferro-manganese containing 1 per cent or more of chromium may not dissolve completely in nitric acid. In this case, dilute the solution after treatment with nitric acid, filter, and fuse the washed residue with a little sodium carbonate. Dissolve the melt in a small volume of nitric acid (sp. gr. 1.135), and add it to the main solution. Treat with potassium nitrate, etc., as previously described.

With low-carbon ferro-manganese or manganese metal dissolve 0.2500 g. of the sample in 250 cc. of nitric acid (sp. gr. 1.135) in a covered 750-cc. Erlenmeyer flask and boil the solution gently for a few minutes to expel oxides of nitrogen. Treat with bismuthate and sulfurous acid as described above.

With silico-manganese (60 to 70 per cent of manganese) and manganese-silicon (20 to 25 per cent of manganese) treat 0.3000 g. of the former and 1.000 g. of the latter with 5 cc. of hydrofluoric acid in a large platinum dish equipped with a gold or platinum cover. (Some spiegeleisens are completely soluble in nitric acid in which

[1] See p. 98.

case the sample can be dissolved directly in 250 cc. of nitric acid (sp. gr. 1.135). When the reaction moderates, add nitric acid (sp. gr. 1.42) a few drops at a time until the sample has completely dissolved. Add 10 cc. of nitric acid (sp. gr. 1.42) and evaporate to a syrupy consistency. Again add 10 cc. of nitric acid (sp. gr. 1.42) and evaporate as before to expel hydrofluoric acid. Dissolve the residue in nitric acid (sp. gr. 1.135), transfer to a 750-cc. Erlenmeyer flask, dilute with nitric acid (sp. gr. 1.135) to 250 cc. and treat as described before with sodium bismuthate, etc.

(*B*) *Oxidation of Manganese*

If the preceding directions have been followed, the manganese will be present in a concentration of approximately 0.001 g. per 1 cc. of nitric acid (sp. gr. 1.135). This concentration of manganese and nitric acid, together with a temperature of 10 to 15° C. are the conditions which insure maximum stability of the permanganic acid, formed as further described. To insure complete oxidation of the manganese to permanganic acid, it is essential that the sodium bismuthate (80 per cent of $NaBiO_3$) be used in the ratio of at least 26 g. to every gram of manganese in solution. Add approximately 7.0 g. of sodium bismuthate (80-per-cent) to the flask, *agitate briskly* for one minute, dilute with 250 cc. of cold water, and filter immediately on a layer of acid-washed asbestos supported on a 2-in. alundum or perforated porcelain plate resting in a large glass funnel. The filter can be washed free from manganese more readily if not allowed to run dry during the filtering and washing. Wash the filter and residue with cold 3-per-cent nitric acid until the washings are entirely colorless and immediately treat the filtrate and washings as described in the following paragraph.

Weigh accurately 9.000 g. of ferrous ammonium sulfate, taken from a bottle of the well-mixed salt, and add to the filtered solution of permanganic acid. Stir briskly and as soon as the reduction is complete and the salt has dissolved, titrate the excess of ferrous salt with 0.1 *N* potassium permanganate. The manganese value of the ferrous salt is obtained in the following manner:

Two hundred and fifty cubic centimeters of nitric acid (sp. gr. 1.135) contained in a 750-cc. Erlenmeyer flask are treated with approximately 2.0 g. of sodium bismuthate and the solution is vigorously agitated for one minute, diluted with 250 cc. of cold water, and immediately filtered on a layer of acid-washed asbestos as previously described. Five (5.000) grams of the ferrous ammonium sulfate are added to the filtrate and the resulting solution is immediately titrated

with 0.1 N $KMnO_4$ until a faint pink color appears. The excess of permanganate used to cause an end point color must be determined by watching the color in another beaker containing the same amounts of acid and cold water.

NOTES

1. Chromium when less than 2 per cent does not cause any interference if the reactions are rapidly done in cold solutions. Larger amounts interfere to some extent and should be separated prior to the final oxidation with bismuthate.

2. The gravimetric determination of manganese by the pyrophosphate method will give high values if the precipitate of manganese ammonium phosphate is allowed to stand more than ten hours before filtration.

3. Volumetric methods such as Volhard's, Ford-Williams' and Julian's do not permit the use of a theoretical titre. Each necessitates the use of an empirical titre obtained by titration of material of known manganese content which has been carried through all steps of the method.

DETERMINATION OF CARBON BY THE DIRECT COMBUSTION METHOD

See the Determination of Carbon in Ferro-Chromium by the Direct Combustion Method.[1] A furnace temperature of 1050 to 1075° C. suffices, and high-silicon material should be covered with 1 or 2 g. of ingot iron.

DETERMINATION OF PHOSPHORUS BY THE ALKALIMETRIC METHOD

SOLUTIONS REQUIRED

Dilute Nitric Acid (sp. gr. 1.135).—Mix 300 cc. of HNO_3 (sp. gr. 1.42) and 700 cc. of distilled water.

Ammonium Molybdate.—See the Determination of Phosphorus in Ferro-Vanadium by the Modified Molybdate-Magnesia Method.[2]

Standard Sodium Hydroxide.—Dissolve 6.5 g. of purified NaOH in 1000 cc. of distilled water, add a slight excess of a 1-per-cent solution of $Ba(OH)_2$, let stand for 24 hours, decant the liquid and standardize against Bureau of Standards Standard Benzoic Acid as follows: Dissolve 0.6 g. of benzoic acid in 15 cc. of alcohol in a small Erlenmeyer flask and titrate in the cold with phenolphthalein indicator,

[1] See p. 101.
[2] See p. 104.

taking precautions to prevent access of carbon dioxide from the air during titration. The ratio of phosphorus to sodium hydroxide is taken as 1 to 23 in calculating the value of the sodium hydroxide. Protect the solution from carbon dioxide with a soda-lime tube.

Standard Nitric Acid.—Mix 10 cc. of HNO_3 (sp. gr. 1.42) and 1000 cc. of distilled water. Titrate the solution against the standard hydroxide solution, using phenolphthalein as an indicator. Adjust the solution so that it is equivalent to the sodium hydroxide solution.

Dilute Ammonium Hydroxide (sp. gr. 0.96).—Dilute 400 cc. of NH_4OH (sp. gr. 0.90) with 600 cc. of water. The strength of this solution should be established by titration or test with a hydrometer.

METHOD

Dissolve 2.000 g. of the sample in 70 cc. of nitric acid (sp. gr. 1.42), evaporate to dryness and bake for two or three hours at 120° C. High-silicon alloys may require the addition of a little hydrofluoric acid for complete decomposition. In this case, several evaporations with 15-cc. portions of nitric acid (sp. gr. 1.42) should be made to remove hydrofluoric acid before the baking treatment. Moisten the residue with 25 cc. of dilute hydrochloric acid (1:1), digest a few minutes,[1] add 75 cc. of water, heat until the salts are dissolved and then filter to remove silica. Evaporate the filtrate to a syrupy condition, add 15 cc. of nitric acid (sp. gr. 1.42) and again evaporate to a small volume. Again add 15 cc. of nitric acid (sp. gr. 1.42) and evaporate to a small volume. Add 100 cc. of nitric acid (sp.gr. 1.135), heat to boiling and add a 1.5-per-cent solution of potassium permanganate until oxides of manganese are precipitated. Dissolve the oxides of manganese by addition of sulfurous acid and boil to expel the oxides of nitrogen. To the cold solution add 40 cc. of NH_4OH (sp. gr. 0.96) and 50 cc. of ammonium molybdate solution and shake for 5 minutes. Allow to settle for 10 minutes; for material containing less than 0.02 per cent of phosphorus, allow to stand 30 minutes. Filter and wash 10 times with a 1-per-cent solution of KNO_3. Return the precipitate and paper to the precipitating vessel, add 1 to 3 cc. in excess of standard NaOH solution and 25 cc. of water, both free from carbon dioxide, and shake or stir until the precipitate is dissolved. Dilute to 100 cc. with water free from carbon dioxide, add 3 drops of a 0.2-per-cent solution of phenolphthalein, and discharge the pink color with standard acid. Finish the titration by adding standard alkali until the reappearance of the pink color. With very

[1] Addition of a little sulfite or sulfurous acid facilitates solution after the baking treatment.

low phosphorus material this titration should be corrected by a blank carried through all steps of the method.

NOTE

If vanadium is present in amounts over 0.08 per cent it should be reduced by ferrous sulfate and sulfurous acid before precipitation with molybdate.

DETERMINATION OF SULFUR BY THE OXIDATION METHOD

Proceed as in the Determination of Sulfur by the Oxidation Method as described in the Standard Methods of Chemical Analysis of Plain Carbon Steel (Serial Designation: A 33) of the American Society for Testing Materials.[1]

NOTES

Routine determinations of sulfur can be made by the evolution method provided concentrated hydrochloric acid (sp. gr. 1.19) is used, preferably in all-glass apparatus of the Pulsifer type.

DETERMINATION OF SILICON BY THE NITRO-SULFURIC ACID METHOD

SOLUTIONS REQUIRED

Nitro-Sulfuric Acid.—Pour 160 cc. of sulfuric acid (sp. gr. 1.84) slowly and with stirring into 660 cc. of water and add 180 cc. of nitric acid (sp. gr. 1.42).

METHOD

Dissolve 0.9344 g. of the sample passing a No. 100 sieve with 50 cc. of the nitro-sulfuric acid in a 250-cc. porcelain casserole and evaporate until copious fumes of sulfuric acid are evolved. After the solution has cooled somewhat add 125 cc. of distilled water and 5 cc. of HCl (sp. gr. 1.19). Heat with frequent stirring until all salts are dissolved and immediately filter on a 9-cm. paper. Wash the precipitate with cold dilute HCl (5:95) and hot water alternately to complete the removal of soluble salts, and finally with hot water until free from acid. Transfer the filter to a platinum crucible, char the paper carefully, and finally ignite with the crucible covered over a blast lamp or in a muffle furnace at 1050 to 1100° C. for at

[1] 1924 Book of A.S.T.M. Standards.

least 10 minutes; cool in a desiccator and weigh. Add sufficient dilute sulfuric acid (1:1) to moisten the silica and then a small amount of hydrofluoric acid. Evaporate to dryness, ignite and weigh. The difference in weights divided by 2 and multiplied by 100 represents the percentage of silicon in the alloy.

NOTE

For silico-manganese and manganese-silicon, fuse 0.4672 g. of the former or 0.2336 g. of the latter with 8 g. of sodium peroxide in a pure iron crucible. Dissolve the fusion and evaporate to fumes of sulfuric acid. When acidifying the solution of the fusion with sulfuric acid it is necessary to add some sulfurous acid to dissolve the manganese dioxide which separates out.

FERRO-CHROMIUM

DETERMINATION OF CHROMIUM BY FUSION WITH SODIUM PEROXIDE

SOLUTIONS REQUIRED

Potassium Permanganate, 0.1 N.—Dissolve 3.2 g. of potassium permanganate in 1000 cc. of distilled water, allow to age for at least ten days and filter through purified asbestos. Standardize the permanganate solution against Bureau of Standards Standard Sodium Oxalate as follows:

In a 400-cc. beaker dissolve 0.25 to 0.30 g. of sodium oxalate in 200 to 250 cc. of hot water (80 to 90° C.) and add 10 cc. of dilute H_2SO_4 (1:1). Titrate at once with the permanganate solution, stirring the liquid vigorously and continuously. The permanganate must not be added more rapidly than 10 to 15 cc. per minute, and the last 0.5 to 1 cc. must be added dropwise, with particular care to allow each drop to fully decolorize before the next is introduced. The excess of permanganate used to cause an end point color must be estimated by matching the color in another beaker containing the same bulk of acid and hot water. The temperature of the solution should not be below 60° C. by the time the end point is reached. One cubic centimeter of 0.1 *N* $KMnO_4$ is equivalent to 0.001734 g. of chromium.

Standard Ferrous Ammonium Sulfate.—Dissolve 43 g. of the salt in 250 cc. of cold water, add 40 cc. of sulfuric acid (1:3), dilute to one liter with distilled water, and mix thoroughly. Large amounts of the solution can be conveniently mixed by means of a stream of carbon dioxide from a cyclinder. As the ferrous ammonium sulfate solution gradually weakens in reducing power it is necessary to standardize it daily or at the same time the ferro-chromium is analyzed.

The solution can be standardized against either potassium dichromate or sodium oxalate.

The first method is somewhat simpler and more satisfactory for routine work as the conditions prevailing in both the standardization and in the analysis are practically identical. No "blank" corrections are afterwards necessary where the amounts of dichromate, ferrous salt and permanganate are approximately those prevailing in the analysis. The second method is employed chiefly for checking the accuracy of the results obtained with potassium dichromate and in cases where only occasional analyses are made and pure dichromate is not readily available.

To prepare pure potassium dichromate, recrystallize at least twice the purest grade of the salt obtainable, dry the crystals at 150° C., grind to a fine powder and again dry at 150° C. to constant weight. Keep the prepared salt in a ground-glass stoppered bottle. Transfer 1.0000 g. of the pure dichromate, which contains approximately the same amount of chromium as an 0.5000-g. sample of 70-per cent ferro-chromium, to an 800-cc. beaker. Dissolve in cold water, acidify with 40 cc. of sulfuric acid (1:3) and dilute with cold water to 500 cc. Add a slight excess of the ferrous sulfate solution, approximately 190 cc., and titrate the excess ferrous salt with 0.1 N permanganate to the first faint permanent darkening of the clear green color.

Example.—The calculations involved are illustrated as follows: In a standardization, 190.0 cc. of the ferrous solution was used and 5.06 cc. of 0.1 N permanganate was required to titrate the excess.

1 cc. of 0.1 N $K_2Cr_2O_7$ contains 0.004903 g. of $K_2Cr_2O_7$

1 g. of $K_2Cr_2O_7$ equals 203.94 cc. of 0.1 N $K_2Cr_2O_7$

then

203.94 + 5.06 = 209.0 cc. of equivalent 0.1 N solution reduced by the ferrous solution.

$$\frac{209.0}{190} = 1.100, \text{ the 0.1 } N \text{ normality factor.}$$

In standardizing through sodium oxalate, it is necessary to add the ferrous ammonium sulfate solution by means of an accurately calibrated pipette or burette instead of an automatic pipette. Transfer 100 cc. of the approximately 0.1 N ferrous solution to a 600-cc. beaker, dilute to 300 cc. with cold 5-per-cent sulfuric acid, add 2 cc. of phosphoric acid and titrate immediately with 0.1 N $KMnO_4$ to a faint permanent pink color. Determine the "blank" on the same volume of water and acids and deduct. When this method is used it is necessary in the actual analysis to determine a "blank" to counteract the influence of the green color of the chromium sulfate upon the permanganate end point. This "blank" can be determined in the solution used in the analysis and is obtained as described later.

METHOD

Transfer 0.5000 g. of the sample of ferro-chromium (dried at 105 to 110° C.) to a 30-cc. heavy-walled porcelain crucible. Samples of low-carbon ferro-chromium should be crushed to pass through a No. 40 screen, while high-carbon ferro-chromium and chromium metal should be reduced to pass a No. 100 screen. Add approximately 8 g. of sodium peroxide and thoroughly mix the contents of the crucible with a small rod, being careful to clean the rod, which can be done conveniently by scraping with another rod. Cover the mixture with 1 to 2 g. of sodium peroxide. Carefully fuse the contents of the covered crucible, preferably in an electric muffle heated to 600 to 700° C. Fusion for five minutes at a low red heat after the mass has melted will insure complete decomposition. When the charge has melted, the crucible should be given a slight rotary motion to stir up any unattached particles.

Place the cooled crucible and cover in a 600-cc. beaker and dissolve the contents in 200 cc. of water. When the melt has dissolved, remove the crucible and cover, add 1 g. of sodium peroxide and boil the solution ten minutes to completely decompose the excess of peroxide.

Allow the precipitate to settle and filter through a pad of ignited asbestos, preferably in a Büchner funnel. An asbestos rather than a paper filter should be used, for the alkaline solution extracts reducing matter from paper which would cause low results for chromium. Thoroughly wash the precipitate with hot water, and at no time allow the precipitate to run dry during the filtering and washing. If desired, the precipitate can be subsequently dissolved in dilute sulfuric acid and tested for chromium by adding a slight excess of sodium peroxide; the amount of chromium retained is negligible if the precipitate is filtered and washed as directed.

Cool the filtrate, acidify with 50 cc. of sulfuric acid (1:1), again cool to room temperature, transfer to an 800-cc. beaker, dilute with cold water to 500 cc. and add a measured excess of ferrous ammonium sulfate. The point at which reduction is complete can be told by changes in the deep green color which is developed. The ferrous salt can be added either as an approximately 0.12 *N* solution or in weighed portions from a bottle of the well-mixed salt. If the solution of ferrous salt is used, approximately 180 cc. will be required for a 70-per-cent alloy; if the salt is added, 8 g. will usually be sufficient. Titrate the excess of ferrous salt with standard 0.10 *N* potassium permanganate. The first faint permanent darkening of the clear green color is taken as the end point, which is quite sharp and well defined to the practiced eye. If the ferrous salt has been standardized

against dichromate, subtract the required number of cubic centimeters of 0.1 N permanganate from the number of cubic centimeters of 0.1 N ferrous solution (obtained by multiplying the number of cubic centimeters of the approximately 0.1 N ferrous solution by the 0.1 N normality factor). The difference represents the number of cubic centimeters of 0.1 N ferrous solution which multiplied by 0.001733 g. gives the number of grams of chromium present.

Where the ferrous solution has been standardized against sodium oxalate, it is necessary to subtract a "blank" from the permanganate titration. This "blank" is obtained as follows: When the end point is reached and the readings have been noted, add about 5 cc. in excess of the ferrous solution and oxidize the excess of ferrous iron by adding 8 cc. of a 15-per-cent solution of ammonium persulfate. *Stir the liquid vigorously for one minute.* Carefully add 0.1 N permanganate to the solution with constant stirring until the same end point is obtained as before. The volume of permanganate required, usually 0.2 to 0.4 cc., constitutes the "blank" to be deducted from the volume of the permanganate titration. For example, in the determination of chromium in an 0.5000-g. sample, 190.0 cc. of a 0.11 N ferrous solution and 11.00 cc. of an 0.1 N permanganate (11.40 – 0.4 cc. blank) were employed.

The calculations are as follows:

$$190.0 \times 1.100 = 209.0 \text{ cc. of } 0.1\ N \text{ ferrous solution}$$
$$209.0 - 11.0 = 198.00 \text{ cc.}$$
$$198.0 \times 0.001734 \times 2 \times 100 = 68.65 \text{ per cent of chromium}$$

The above calculations are given for exactly 0.1 N permanganate; if the permanganate is not 0.1 N, the volume used must be converted to the equivalent of 0.1 N by multiplying by the proper factor.

DETERMINATION OF CARBON BY THE DIRECT COMBUSTION METHOD

Determine carbon by the direct combustion method described in the Standard Methods of Chemical Analysis of Plain Carbon Steel (Serial Designation: A 33), with the following modifications:

1. Ferro-chromium alloys require a furnace temperature of 1125 to 1150° C. in order to yield all of the carbon in the material.

2. The determination is made as given in the reference except that the weighed sample of ferro-chromium, 0.5000 g. for 4 to 8-per-cent carbon and 3 to 1 g. for 0.10 to 2.00-per-cent carbon, is mixed

with 1 g. of copper oxide and covered with 2 g. of pure ingot iron drillings in order to facilitate complete combustion.

3. On account of the small weight of sample used, the blanks are important and must be carefully determined.

DETERMINATION OF SILICON

Low-carbon ferro-chromium and chromium metal can be decomposed by means of dilute sulfuric acid, but the high-carbon alloy has to be fused with sodium peroxide. For low-carbon ferro-chromium, transfer 0.9344 g. (a double factor weight) to a 250-cc. porcelain casserole, add 60 cc. of sulfuric acid (1:4) and warm the covered casserole gently until the reaction is complete. For high-carbon ferro-chromium, transfer 0.9344 g. of the sample passing a No. 100 sieve to a 30-cc. pure iron crucible made from No. 20 gage (0.032 in. thickness) ingot iron. Add approximately 8 g. of sodium peroxide, mix thoroughly and fuse as directed under the determination of chromium. When the fusion has solidified, tap the covered crucible, while still warm, on an iron plate to loosen its contents in a solid cake which is then transferred to a 250-cc. casserole. Rinse the crucible with a little water, transfer the solution to the casserole, cover, and carefully add 60 cc. of sulfuric acid (1:4) after which the cover glass is rinsed and removed.

Evaporate the solution obtained by either of the above procedures until fumes of sulfuric acid are given off. If chromium sulfate solutions are fumed at too high temperatures or for any great length of time or with too little free acid present, considerable difficulty will occur in dissolving the salts. The fuming should, therefore, be made at a temperature not much higher than that required for evolving the acid. Allow the casserole and contents to partly cool (if permitted to become entirely cold, difficulty may be experienced in getting chromium salts to dissolve), add 100 cc. of warm water while stirring to prevent the residue from caking on the bottom and boil the solution gently for several minutes to dissolve all salts. Filter, washing the paper several times with cold dilute hydrochloric acid (2-per-cent), and then with warm water. Transfer the filter and contents to a platinum crucible, char the paper, carefully ignite to remove carbon, and then heat the covered crucible over a blast lamp or in a muffle furnace at 1050 to 1100° C. for at least 15 minutes. Cool in a desiccator and weigh. Add sufficient dilute sulfuric acid (1:1) to moisten the silica and then a small amount of hydrofluoric acid. Evaporate to dryness, ignite and weigh. The difference in weights represents silica which contains 46.72 per cent of silicon.

NOTE

Crucibles made from pure ingot iron contain only traces of silicon and the amount of silica present in the sodium peroxide used is usually negligible, but "blanks" should be run on new lots of crucibles and of peroxide. All nickel crucibles, even the German "Rein" nickel, contains appreciable amounts of silicon.

FERRO-VANADIUM

DETERMINATION OF VANADIUM BY THE AMMONIUM PERSULFATE METHOD

SOLUTIONS REQUIRED

Ammonium Persulfate (15 per cent).—Make up as needed by dissolving 15 g. of the salt in 100 cc. of distilled water. This salt should be 95-per-cent pure and its purity tested by adding 30 cc. of a standard ferrous sulfate solution to 0.3 g. of the persulfate, then 200 cc. of hot water and titrating the excess ferrous salt with potassium permanganate. One cubic centimeter of 0.1 N ferrous solution equals 0.01141 g. of $(NH_4)_2S_2O_8$.

Potassium Ferricyanide Indicator.—Dissolve 0.1 g. of the salt in 100 cc. of water. This solution should be made up fresh daily.

Standard Ferrous Ammonium Sulfate, approximately 0.1 N.—See the Determination of Chromium in Ferro-Chromium.[1]

Potassium Permanganate, 0.1 N.—See the Determination of Chromium in Ferro-Chromium.[1] One cubic centimeter of 0.1 N $KMnO_4$ is equivalent to 0.005096 g. of vanadium in the following method.

METHOD

Treat 0.5000 g. of the alloy, dried at 105 to 110° C., in a 250-cc. covered beaker with a mixture of 60 cc. of sulfuric acid (1:3) and 25 cc. of nitric acid (1:1). If the alloy fails to decompose readily because the silicon content is high, it may be necessary to add a small amount of hydrofluoric acid. In this event, a 200-cc. platinum dish should be substituted for the beaker. When the reaction has abated, rinse the cover glass and evaporate the solution until copious fumes of sulfuric acid are evolved. Cool, add 100 cc. of water and heat until the salts are dissolved. Transfer the solution to an 800-cc. beaker, dilute to 400 cc. and to the cooled solution add an approximately 0.1 N solution of potassium permanganate until a strong pink color has developed which remains permanent for thirty seconds.

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[1] See p. 98.

Next reduce the vanadium by the use of an approximately 0.1 *N* solution of ferrous ammonium sulfate added until a drop of the solution withdrawn from the beaker and placed on a drop of potassium ferricyanide indicator shows ferrous iron present—about 35 cc. of 0.1 *N* ferrous solution. Add 5 cc. of the ferrous ammonium sulfate in excess, stir the solution at least one minute, oxidize the excess of ferrous sulfate with 8 cc. of 15-per-cent ammonium persulfate and stir the solution vigorously for *one minute.* Titrate the solution with 0.1 *N* potassium permanganate solution, added with constant stirring until a faint pink color appears which remains permanent for 30 seconds. Determine the "blank" by dissolving 0.4 g. of "ingot iron" in 60 cc. of H_2SO_4 (1:3) and 5 cc. of nitric acid (sp. gr. 1.42), and putting the solution through all of the operations of the analysis. If the alloy contains an appreciable amount of chromium, a weight of pure $K_2Cr_2O_7$ equivalent to the amount of chromium in the sample is dissolved in the acids with the ingot iron.

DETERMINATION OF CARBON

See the Determination of Carbon in Ferro-Chromium by the Direct Combustion Method.[1] A furnace temperature of 1050 to 1100° C. will suffice for ferro-vanadium and complete combustion except in very high-silicon material can be secured without the addition of ingot iron and copper oxide.

DETERMINATION OF PHOSPHORUS BY THE MODIFIED MOLYBDATE-MAGNESIA METHOD

Solutions Required

Ammonium Molybdate.—

Solution No. 1.—Place in a beaker 100 g. of 85-per-cent molybdic acid, mix thoroughly with 240 cc. of distilled water, add 140 cc. of NH_4OH (sp. gr. 0.90), filter, and add 60 cc. of HNO_3 (sp. gr. 1.42).

Solution No. 2.—Mix 400 cc. of HNO_3 (sp. gr. 1.42) and 960 cc. of distilled water.

When the solutions are cold, add Solution No. 1 to Solution No. 2, stirring constantly, then add 0.1 g. of ammonium phosphate dissolved in 10 cc. of distilled water, and let stand at least 24 hours before using.

[1] See p. 101.

Magnesia Mixture.—Dissolve 50 g. of $MgCl_2 \cdot 6H_2O$ and 200 g. of NH_4Cl in 1000 cc. of distilled water containing a few cubic centimeters of hydrochloric acid.

METHOD

Transfer 4 g. of the sample to a platinum dish of 150 to 200-cc. capacity, add 70 cc. of dilute HNO_3 (5:6) and heat the covered dish until vigorous action ceases. Add 1 or 2 cc. of hydrofluoric acid, stir the solution, evaporate to dryness, moisten with 10 cc. of nitric acid (sp. gr. 1.42) and again evaporate to dryness. Moisten the cooled residue with 10 cc. of HNO_3 (sp. gr. 1.42), add 50 cc. of water and wash the contents of the dish into a 400-cc. beaker, rubbing the dish well with a policeman. Remove the last traces of adhering salts with 5 cc. of HCl (sp. gr. 1.19) and hot water. Add 10 cc. of HNO_3 (sp. gr. 1.42) and evaporate to a syrupy consistency. Cool, add 25 cc. of HNO_3 (sp. gr. 1.42) 60 cc. of water, 1 g. of boric acid (to take care of any residual fluoride) and heat gently until solution occurs. Filter through a rapid paper into a 300-cc. Erlenmeyer flask and wash with hot water. Heat the solution to boiling, add about 12 cc. of potassium permanganate solution (1.5-per-cent) and continue the boiling until manganese dioxide precipitates. Dissolve the precipitate by the addition of sufficient sulfurous acid added dropwise. About 1 cc. usually suffices. Boil for a few minutes to completely expel oxides of nitrogen. Add to the cold solution 15 cc. of ammonium hydroxide (sp. gr. 0.90) and cool the solution to 15° C.

Next reduce the vanadium in the cold (15° C.) solution by ferrous sulfate added in approximately 1.5 g. excess. The use of a 50-per-cent solution of the salt furnishes a convenient way of adding the necessary amount. One gram of quinquevalent vanadium requires 5.5 g. of $FeSO_4 \cdot 7H_2O$ for reduction to the quadrivalent state. A 4-g. sample of 30-per-cent vanadium alloy, including an excess of 1.5 g., would require 8.1 g. of ferrous sulfate. Too large an excess of ferrous sulfate should be avoided. The addition of ferrous sulfate will change the color of the solution to a deep blue. Add 2 drops of a saturated solution of sulfurous acid, 100 cc. of molybdate reagent, shake for 10 minutes and allow to stand at 15° C. for 4 to 6 hours, or preferably overnight. It is well to let the solution stand in a refrigerator. Filter on a paper of close texture, keeping as much of the precipitate in the flask as possible, and wash the flask and the precipitate with not more than 50 cc. of cold, dilute nitric acid (1:99). Set the filtrate and washings aside after thorough mixing and see whether further separation of phosphomolybdate occurs. Dissolve the precipitate on the

filter in 20 cc. of dilute ammonium hydroxide (1:1) to which have been added 2 g. of citric acid, catch the solution in the original flask containing the bulk of the yellow precipitate and finally wash the filter several times with dilute hydrochloric acid (5:95). If the ammoniacal solution of the phosphomolybdate is not clear at this point, heat it to boiling, filter through the same paper, and wash the paper with hot water. In this case, and whenever appreciable amounts of such elements as titanium or zirconium are known to be present in the original material, preserve the paper and ignite it in a small platinum crucible with the paper reserved below.

Render the ammoniacal solution acid with hydrochloric acid, add 20 cc. of magnesia mixture and then ammonium hydroxide (sp. gr. 0.90) slowly until a crystallin precipitate appears, and then finally an excess of 3 to 5 per cent by volume. The volume of the solution at this point should not exceed 100 cc. Allow the solution to stand in a cool place for 4 to 6 hours, or preferably overnight.

Filter, keeping as much of the precipitate as possible in the flask, and wash the flask and the precipitate moderately with dilute ammonium hydroxide (1:20). Set the filtrate and washings aside after thorough mixing and see whether further precipitation occurs. Dissolve the precipitate on the filter in 25 cc. of dilute hydrochloric acid (1:1), catching the solution in the original flask containing the bulk of the precipitate. Wash the filter thoroughly with dilute hydrochloric acid (5:95). In case the original material contains appreciable amounts of the elements mentioned above (titanium or zirconium), add the paper to the one reserved above, ignite, fuse any residue with as little carbonate as possible (not over 0.5 g.), extract the cooled melt with hot water, filter, and add the water solution to the hydrochloric acid solution. Transfer the solution to a 200-cc. beaker. In case the amount of arsenic is known to be very small, add 0.5 to 1 g. of ammonium bromide and boil the hydrochloric acid solution to a volume of 5 to 10 cc. Where large amounts of arsenic were originally present, treat with hydrogen sulfide, digest, filter, and wash with acidulated hydrogen sulfide water. Boil to expel hydrogen sulfide and to reduce the volume to 50 to 75 cc. Add 0.1 to 0.2 g. of citric acid, 2 to 3 cc. of magnesia mixture, make ammoniacal as above, and allow to stand in a cool place for 4 to 24 hours.

Filter, wash with dilute ammonium hydroxide (1:20), and ignite the precipitate carefully and at as low a temperature as possible until the carbon has been destroyed and the residue is white. Finally, ignite at approximately 1000° C. to constant weight. Dissolve the ignited residue in 5 cc. of dilute nitric acid (1:1) and 20 cc. of water.

Filter, wash the residue with hot water, ignite and weigh. Treat with a few drops of hydrofluoric acid, evaporate to dryness, ignite and re-weigh. Subtract any loss of weight from the original weight of the pyrophosphate, and calculate on the basis of this purified magnesium pyrophosphate.

DETERMINATION OF SULFUR BY THE OXIDATION METHOD

Transfer 4.57 g. of the sample to a 600-cc. beaker and dissolve it in 75 cc. of HNO_3 (sp. gr. 1.42). The reaction is apt to be very rapid and the acid should be added in small portions to the covered beaker which is preferably kept cooled by immersion in ice water. Effect complete solution by gentle warming if necessary.

Add 30 cc. of HCl (sp. gr. 1.19) and evaporate to dryness on the steam-bath. Add 30 cc. more of HCl (sp. gr. 1.19), again evaporate to dryness, and bake for 30 minutes at 105° C. Moisten the residue with 30 cc. of HCl (sp. gr. 1.19) and evaporate on the steam-bath to a volume of 5 cc. Add 5 cc. of HCl (sp. gr. 1.19), dilute to 150 cc. and filter. Wash with warm water.

Adjust the volume of the solution to 250 cc. which should now contain about 3 to 4 per cent by volume of HCl. Warm to 75° C., add 30 cc. of $BaCl_2$ (10-per-cent), and allow to stand for 24 to 40 hours.

Filter, with as little transfer of the precipitate as possible, through a paper of close texture. Wash the paper two or three times with cold 1-per-cent HCl and seven times with warm water. Ignite the paper and contents in a platinum crucible. Treat the ignited residue with 5 cc. of HCl (sp. gr. 1.19) and transfer the resulting solution to the original beaker containing most of the precipitate of $BaSO_4$. Digest the combined portions of $BaSO_4$ so as to dissolve any barium vanadate. Adjust to 150-cc. volume and 2-per-cent acidity, add 8 cc. of 10-per-cent $BaCl_2$ solution and allow to stand for 24 hours. Filter on a paper of close texture, wash three times with cold 1-per-cent HCl and 10 times with warm water. Ignite the paper in platinum, cool in a desiccator and weigh. A blank should be carried through all steps of the determination. The corrected weight of the ignited barium sulfate multiplied by three is equal to the percentage of sulfur.

NOTES

If desired, the washings can be kept separate and examined for $BaSO_4$ as follows: Evaporate the washings to dryness. Dissolve the slight residue in 50 cc. of hot dilute HCl (2:98), add 2 cc. of $BaCl_2$ solution (10-per-cent), digest at 70 to 80° C. for two hours, avoiding any undue evaporation. Filter on a small paper of close texture and wash as above. The recovery of $BaSO_4$, which is ordinarily obtained, represents approximately 0.001 to 0.002 per cent of sulfur.

In high-silicon alloys the residue from the initial acid digestion should be evaporated with hydrofluoric and nitric acids, fused with sodium carbonate and the water extract of the melt added to the main solution.

DETERMINATION OF SILICON
BY THE
NITRO-SULFURIC METHOD

SOLUTIONS REQUIRED

Nitro-Sulfuric Acid.—Pour 160 cc. of sulfuric acid, (sp. gr. 1.84) slowly and with stirring into 660 cc. of water, and add 180 cc. of nitric acid (sp. gr. 1.42).

METHOD

Dissolve 0.9344 g. of the sample passing a No. 100 sieve of low or medium-silicon ferro-vanadium in 40 cc. of the nitro-sulfuric acid in a 250-cc. porcelain or platinum dish and evaporate the solution until "copious" fumes of sulfuric acid are evolved.

Ferro-vanadium containing 4 per cent or more of silicon is not completely soluble in nitric acid and sulfuric acid. In this case, fuse 0.9344 g. of the alloy, thoroughly mixed with 8 g. of sodium peroxide, in a 30-cc. crucible of pure iron. Five minutes heating at a dull red heat after the contents of the crucible have melted will insure complete decomposition of the sample. Allow the fusion to solidify, but before it has entirely cooled tap the covered crucible on an iron plate which will loosen the contents in a solid cake. Transfer the melt to a 250-cc. porcelain casserole, rinse the crucible with warm water and cautiously add the solution to the covered casserole. Next add 150 cc. of sulfuric acid (20 per cent by volume) and evaporate to copious fumes of sulfuric acid. Cool the sulfuric acid solution obtained in either procedure, add 125 cc. of water and heat for a few minutes with frequent stirring until all salts are dissolved. Immediately filter on a 9-cm. paper. Wash the precipitate with cold dilute HCl (5:95) and cold water alternately to complete the removal of iron salts, and finally with hot water until free from acid. Transfer the filter to a platinum crucible, char the paper without inflaming and

finally ignite the covered crucible over a blast lamp or in a muffle furnace at 1050 to 1100° C. for at least 15 minutes. Cool in a desiccator and weigh. Add sufficient dilute H_2SO_4 (1:1) to moisten the silica and then a small amount of hydrofluoric acid. Evaporate to dryness, ignite and weigh. The difference in weights divided by 2 and multiplied by 100 represents the percentage of silicon in the alloy.

DETERMINATION OF ALUMINUM BY THE ETHER-HYDROCHLORIC ACID METHOD[1]

Dissolve 2 g. of the sample in 25 to 30 cc. of dilute nitric acid (sp. gr. 1.135) (Note 1). When action ceases, add 5 cc. of hydrochloric acid and evaporate to dryness. Add 10 cc. of hydrochloric acid, evaporate to dryness and bake the residue at a moderate temperature. Dissolve the residue in 10 cc. of hydrochloric acid, add 30 cc. of water, boil and filter. Wash the residue with hot dilute hydrochloric acid (1:9) followed by water (Note 2).

Evaporate the solution to dryness on a steam-bath, add 20 cc. of hydrochloric acid and warm gently until complete solution is obtained. Cool the solution in ice water and pass hydrochloric acid gas into it to saturation. Add 20 cc. of ether and continue passing the gas into the cold solution until it is again saturated. Allow the mixture to stand for several hours, keeping it cold by immersing the container in ice water. If the solution becomes warm or if it is not allowed to stand, the separation of hydrated aluminum chloride is not quite complete.

Filter through asbestos (small Bückner-asbestos pad washed once with cold ether-hydrochloric acid mixture immediately before use), wash, by decantation, with a cold mixture of equal parts of strong hydrochloric acid and ether which has been saturated with hydrochloric acid gas. The aluminum chloride retains a little vanadium.

Dissolve the precipitated aluminum chloride in the least possible amount of dilute hydrochloric acid (1:9) back into the original beaker, from which all of the precipitate need not have been removed. Evaporate the solution to near dryness on a steam-bath, add 10 cc. of strong hydrochloric acid and pass hydrochloric acid gas into the solution to saturation, add 15 cc. of ether and continue passing hydrochloric acid gas to saturation, keeping the solution cold by

[1] Gooch-Havens, *Am. Journal of Science*, Vol. 4, pp. 11–416.

means of ice water. Let stand several hours, filter on asbestos and wash with the ether - hydrochloric acid mixture as before.

Dissolve the aluminum chloride in dilute hydrochloric acid (1:9) into a 250-cc. beaker, add a few grams of ammonium chloride, make faintly alkaline with ammonia, boil, filter (using paper pulp), wash with hot water, dry, ignite and weigh as Al_2O_3.

According to published data, this method gives a complete separation of aluminum from iron, beryllum, zinc, copper, mercury and bismuth. A little vanadium is carried down in the first separation, but two chloride precipitations seem to give a practically complete separation. The proportion of vanadium retained in the final alumina is hardly appreciable when working on a 35-per-cent vanadium alloy.

The effect of chromium is not known; presumably some of it would be included in the Al_2O_3. This could be corrected for by fusing the Al_2O_3 with sodium peroxide and estimating the Cr_2O_3 by color.

NOTES

1. Dilute nitric acid (sp. gr. 1.135) is recommended for solution. While this requires rather long digestion at steam-bath temperature, solution seems to be quite complete even when the alloy is high in silicon. In some cases where silicon is very high it is necessary to use a little hydrofluoric acid with the nitric acid. In this case solution should be effected in a platinum dish, the solution evaporated to dryness and the dry residue transferred to a beaker before treatment with hydrochloric acid. A little dilute hydrochloric acid may be used to loosen any oxides that adhere to the dish.

2. The siliceous residue may retain alumina; it should be ignited in platinum. Silica should be expelled by $HF + H_2SO_4$ and the residue fused with $KHSO_4$. After dissolving the fusion in water plus a little HCl, the liquid is made faintly alkaline with ammonia and boiled. Any iron and alumina precipitate is removed by filtration, washed with water, dissolved in a little hot dilute HCl and added to the solution of the alumina obtained after the first ether - hydrochloric acid separation.

FERRO-TUNGSTEN AND TUNGSTEN METAL

DETERMINATION OF TUNGSTEN

SOLUTIONS REQUIRED

Cinchonine Solution.—Dissolve 125 g. of cinchonine in a mixture of 500 cc. of HCl (sp. gr. 1.19) and 500 cc. of distilled water.

Cinchonine Wash Solution.—Dilute 30 cc. of the above solution to one liter.

Treat 1 g. of the material passing a No. 100 sieve in a large (60-cc.) platinum crucible with 5 cc. of HF; add HNO_3 drop by drop until the metal dissolves. Add 3 to 4 cc. of H_2SO_4 and evaporate on a steam-bath until $HNO_3 + HF$ is expelled. Shake gently over a

small Bunsen flame, or heat slowly in a sheet iron "radiator" (described by Hillebrand)[1] until H_2SO_4 fumes strongly. Cool, transfer to a 250-cc. beaker with water, finally wiping the crucible with a little filter paper. A little WO_3 sticks to the crucible; it can not be removed by wiping. Reverse the crucible. Dilute the contents of the beaker to about 150 cc. with water, add 10 cc. of hydrochloric acid (sp. gr. 1.19) and boil for a few minutes. Remove from the source of heat, add 10 cc. of cinchonine solution and digest the solution at 80 to 90° C. with occasional stirring, for 30 to 45 minutes.

Add some paper pulp to the solution, stir the solution and when the tungsten precipitate has settled, filter on a 9-cm. paper containaing a little "ashless" filter pulp. Wash thoroughly with hot cinchonine solution and finally several times with warm 1-per-cent hydrochloric acid. Gently ignite the filter and residue of crude WO_3 in the crucible in which it was originally treated until the carbon is consumed. Add a few drops of nitric acid and dry in the radiator. Ignite the covered crucible for 5 minutes in the full heat of a Bunsen burner, cool, and weigh. Ignitions may be made at about 750° C. in an electric muffle. As WO_3 is slowly but steadily volatilized at temperatures above 750° C., serious error may result from continued ignition of the tungsten trioxide at the temperature that is specified. Add about 5 g. of Na_2CO_3 and fuse, running the fusion around the side of the crucible to remove all WO_3. Dissolve the fusion in hot water, add alchohol, heat, filter and wash with hot water. Place the filter in the crucible and ignite; add a little Na_2CO_3 and fuse again. Dissolve the fusion in water, filter and wash *very* thoroughly with hot water to remove the last traces of Na_2CO_3; ignite in the same crucible as at first, cool, and weigh. The difference between the weight of crucible plus residue and the weight of crucible plus impure tungstic oxide is $WO_3 \times 0.7931 = W$.

NOTES

At the best, direct determination of tungsten in high-grade metal is subject to inherent errors. Accuracy within 0.2 per cent is all that can be expected by any method.

DETERMINATION OF CARBON

See the Determination of Carbon in Ferro-Chromium by the Direct Combustion Method.[2] The alloy ground to pass a No. 100 sieve and mixed with 2 g. of pure iron is burned in an electric furnace heated at 1050 to 1100° C. A "blank" on the ingot iron must be determined and deducted.

[1] U. S. Geological Survey *Bulletin No. 700*, p. 33.
[2] See p. 101.

DETERMINATION OF PHOSPHORUS

Treat 1 g. of the finely powdered sample in a platinum dish, fitted with a gold cover, with 15 cc. of HNO_3 (sp. gr. 1.42), add 3 cc. of HF and warm gently. When action subsides add 3 cc. more of HF. After action subsides, boil, remove cover and if decomposition is not complete add more HF and boil again. When solution is complete, wash off the cover and evaporate at a low heat to a volume of about 10 cc., then add 3 or 4 drops of concentrated permanganate solution and continue evaporation until crusts of WO_3 begin to form at the edges, that is to a volume of about 6 cc. Add 5 cc. of H_2SO_4 and evaporate on the stove at a low heat until HF and HNO_3 are expelled and H_2SO_4 fumes are given off. (Strong heat causes spattering and also causes hard over-baked crusts to form on the bottom of the dish which resist subsequent treatment.) Cool, add 25 cc. of water and boil (by agitating over a Bunsen flame) until all soluble salts are dissolved. Discharge pink color, due to excess permanganate, by adding sulfurous acid drop by drop. The pink color may not be very evident but the SO_2 is added even so to reduce higher oxides of manganese. Boil for a minute or two after adding the SO_2. Add 1.20 g. of pure tartaric acid and, when this is dissolved and the solution is cooled to a temperature of about 50° C., add 20 cc. of NH_4OH (sp. gr. 0.90) diluted with an equal volume of water. The precipitated tungstic acid should dissolve completely, giving a clear solution. The solution becomes hot from the reaction between H_2SO_4 and NH_4OH. While it is still hot, add 10 cc. of magnesia mixture and transfer it from the platinum dish to a 6-oz. glass-stoppered bottle. Set the bottle in ice water and when it is thoroughly cooled add four or five glass beads about 6 mm. in diameter. Stopper it tightly and shake in an efficient shaking machine for at least ten minutes. The agitation should be violent. The beads aid in starting the formation of the magnesium precipitate; after agitation add 15 cc. of strong NH_4OH and return the bottle to the ice-water tank and put into a refrigerator to stand overnight. Phosphorus separates as magnesium ammonium phosphate free from tungsten but containing possibly basic magnesium compounds. After standing overnight filter the solution through a 9-cm. paper containing a little paper pulp and wash the bottle and paper thoroughly by small additions of ammonia wash water (1 part NH_4OH to 3 parts H_2O). Do not attempt to remove all the precipitate from the bottle but remove the beads to the filter. The precipitate consists of magnesium ammonium phosphate and arsenate together with silica and other impurities. Tin,

tungsten, molybdenum, vanadium and titanium are eliminated by precipitation with magnesia mixture in the presence of tartaric acid. Dissolve the magnesia precipitate in hot dilute HCl and evaporate to a small volume, say 7 or 8 cc. Add 10 cc. of strong HCl and 0.5 g. of KBr and evaporate to dryness. Take up with 5 cc. of HNO_3 and evaporate to dryness again. The KBr serves to reduce As^{v} to As'''. The addition of strong HCl and boiling followed by evaporation to dryness eliminates As. Dissolve residue in 60 cc. of HNO_3 (sp. gr. 1.135), filter into a 250-cc. shaking bottle; add 40 cc. of NH_4OH (sp. gr. 0.96), cool to 33° C., add 30 cc. of molybdate solution (Blair) and finish the determination by the alkalimetric method (Handy) described in Blair's Chemical Analysis of Iron.

NOTES

The usual proportions of HNO_3 and HF are reversed in order to provide a constant excess of HNO_3 to oxidize P. The procedure given takes a little longer than when the sample is treated with HF first and HNO_3 is added a little at a time, but the solution is finally complete. It is necessary to keep the platinum dish covered after action begins, as the reaction is somewhat violent.

The platinum dishes recommended are:

8 cm. in diameter at top;
7.8 cm. in diameter at bottom;
4 cm. high.

They have flat bottoms and are wire-rimmed at the top to give additional stiffness. They weigh 58 to 60 g. each and hold about 175 cc. The covers are made of pure gold (for economy), "dished" like a crucible cover to fit the top of the dishes closely. The dishes have small lips to aid pouring. The "tongue" of the cover overlaps the lip. Ordinary round-bottomed dishes may be used, but the manipulation is much more difficult; there is a greater tendency to spattering and danger of local baking or overheating in evaporating to fumes of H_2SO_4. If the separated WO_3 is overheated locally it does not dissolve readily in NH_4OH. In flat-bottomed dishes, the WO_3 is spread in a thin layer and heat is applied evenly all over the bottom.

A good shaking machine should be used. The magnesia precipitate may be started by shaking the bottles by hand, but it is a tiresome job. The solution must be cold, say 8 or 10° C., when shaking begins.

Permanganate solution is added to insure complete oxidation of phosphorus, as in steel analysis. The color of the permanganate gradually fades in the hydrofluoric nitric solution, but after evaporating to fumes and adding water the solution is usually slightly pink.

The amount of tartaric acid is limited to 1.20 g. since ammonium tartrate retards the formation of the magnesia precipitate. Complete precipitation can only be obtained by brisk agitation and by keeping the solution very cold, followed by long standing in a cold place. By this method determinations started at 3 p. m. may be completed by noon the next day.

DETERMINATION OF SULFUR AND SILICON

Solutions Required

Cinchonine Solution.—Thoroughly wash cinchonine reagent with hot water on a Büchner funnel, dry, and dissolve 125 g. of the washed cinchonine in a mixture of 500 cc. of HCl (sp. gr. 1.19) and 500 cc. of distilled water.

Cinchonine Wash Solution.—Dilute 30 cc. of the above solution to one liter.

Barium Chloride Solution.—One hundred grams of $BaCl_2 \cdot 2H_2O$ to 1000 cc.

SULFUR

Fuse 2 g. of the finely powdered metal, intimately mixed with 8 g. of dry Na_2CO_3 and 2 g. of powdered KNO_3, in a large platinum crucible (40 cc.). The fusion is best done over an alcohol blast flame or in an electric muffle to avoid contamination with sulfur contained in illuminating gas. Leach the fusion with 100 cc. of distilled water in a 250-cc. beaker. Filter into a 12-cm. porcelain casserole and wash the residue several times with hot distilled water. The solution contains practically all of the sulfur and most of the tungsten and silica. The residue contains some of each, but not more than traces of sulfur.

Render the liquid acid with 20 cc. of HCl and evaporate to dryness on a steam-bath, thus precipitating tungstic acid. After the residue is dry, heat it to 110 to 120° C. in an oven to dehydrate silica, take up with 5 cc. of HCl and 50 cc. of water, add 3 cc. of a strong cinchonine solution and boil, or heat just short of the boiling point, for 30 minutes. Let the solution stand for at least three hours and preferably overnight. By means of the cinchonine, all WO_3 is rendered insoluble. Filter through an 11-cm. filter paper (free from ash) which has been liberally treated with an emulsion of filter paper pulp and wash the residue with dilute cinchonine solution. Reserve the residue. The filtrate contains all the sulfur; heat it to boiling and add 10 cc. of barium chloride solution, boil for ten minutes, allow it to stand overnight, filter on a 9-cm. filter paper (free from ash), wash repeatedly but not excessively with hot water, ignite in platinum and weigh as $BaSO_4$. If the work is properly carried out every trace of WO_3 is removed from the solutions and there is no danger of including $BaWO_4$ with the $BaSO_4$. If one suspects that the $BaSO_4$ is not quite pure, it should be fused with a little Na_2CO_3, leached with water and filtered; the filtrate should be acidulated with HCl, a few drops of cinchonine solution added and the solution evaporated to dryness on a steam-bath. The dry residue should be dissolved in a little water,

a few drops of HCl added, and any residue filtered off. In the clear filtrate $BaSO_4$ should be again precipitated as before. The weight of $BaSO_4 \times 0.1374 = S$.

"Blanks" on all reagents must be made parallel with the determination; this is important since all sodium carbonate obtainable contains sulfur and there is sometimes more or less of it in cinchonine. All evaporations should be made over steam and the operations conducted in a place free from sulfur gases.

NOTE

Evolution of sulfur in a stream of hydrogen saturated with hydrochloric acid as in Johnson's Method is optional.

SILICON

The residue of tungstic oxide from which the solution for the determination of sulfur was filtered contains most of the silica. The residue from leaching the original fusion contains the rest.

Wash the residue from Na_2CO_3 fusion from the filter paper with a fine jet of water into a small casserole, add an excess of HCl and evaporate to dryness on a steam-bath, heat the dry residue at 110 to 120° C. to dehydrate the silica, drench with hydrochloric acid, dilute to 100 cc. with hot water, heat until salts are in solution, and filter on a small filter paper (free from ash). Wash thoroughly with hot dilute HCl (1:100) and finally with hot water. Reserve the filter.

Wash the residue of tungstic oxide, silica and filter paper pulp from the filter into a small casserole, add 5 cc. of HCl and heat for a few minutes, then filter again through the same paper and wash it *thoroughly* with hot water. This second washing is necessary to remove sodium salts completely. (The original washing sufficed to remove all of the small amount of sulfur present.) Transfer the well-washed filter and residue to a platinum crucible, add the small filter containing the little silica recovered from the Na_2CO_3 fusion residue and ignite both to constant weight in a platinum crucible. On account of the presence of filter paper pulp, the ignited residue is porous and friable; if paper pulp had not been used the WO_3 would be dense and not easily susceptible to the subsequent treatment. Cool the ignited residue of WO_3 and SiO_2 and weigh it. Add 2 or 3 drops of H_2SO_4 and 5 or 6 cc. of pure HF. Digest at a gentle heat for some time, and then slowly evaporate off the HF. When H_2SO_4 fumes are evolved, cool and again add HF, digest as before, evaporate off the HF, ignite gently to expel H_2SO_4 and heat strongly for ten or fifteen minutes. Cool and weigh. The loss in weight after expulsion is SiO_2 which multiplied by 0.4672 equals silicon.

TENTATIVE RECOMMENDED PRACTICE
FOR
CARBURIZING AND HEAT TREATMENT OF CARBURIZED OBJECTS[1]

Serial Designation: A 37 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope.

1. This recommended practice is intended solely for guidance, and is not to be construed as entering in any manner into specifications of the American Society for Testing Materials.

Purpose and General Procedure.

2. Carburizing is intended to increase the carbon content in the surface layers of low-carbon steel objects so they may be made hard and wear-resistant on the outside with a strong, tough, inner portion or core. This type of structure is suitable for parts which are required to stand considerable shock and friction. The process consists in heating the steel in contact with a carbonaceous material for a long time at temperatures above the critical temperature of the steel.

Factors Governing the Process.

3. The primary factors governing the process are:

(*a*) Chemical composition of the steel;

(*b*) The carburizing operation;

(*c*) The hardening operation.

Chemical Composition of Steel.

4. The steel should be selected according to the service requirements. A well-made carbon steel will be satisfactory for most ordinary purposes. The carbon content of simple and alloy steels should preferably be below 0.20 per cent. High manganese above 0.60 per cent is sometimes specified to obtain increased hardness and increased core strength. High phosphorus and sulfur contents improve the machineability but generally indicate a steel of poor quality. These elements are, however, often added intentionally to steels of good quality to improve their machining qualities.

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[1] Criticisms of this Recommended Practice are solicited and should be directed to Mr. J. H. Hall, Secretary of Committee A-4 on Heat Treatment of Iron and Steel, Taylor-Wharton Iron and Steel Co., High Bridge, N. J.

This Recommended Practice, when adopted as standard, will supersede the present Recommended Practice for Heat Treatment of Case-Hardened Carbon-Steel Objects (Serial Designation: A 37 – 14), 1924 Book of A.S.T.M. Standards.

In alloy steels, nickel tends to retard the grain growth at carburizing temperatures, depresses the critical temperature, and allows the steel to be hardened at lower quenching temperatures. Chromium is used alone in percentages up to 1.25 per cent and gives a greater abrasive hardness to the case. S. A. E. Steel No. 3115, containing about 1.25 per cent nickel and 0.60 per cent chromium is recommended for general high grade use. A molybdenum content of about 0.25 per cent or combined with about 1.75 per cent of nickel is used with

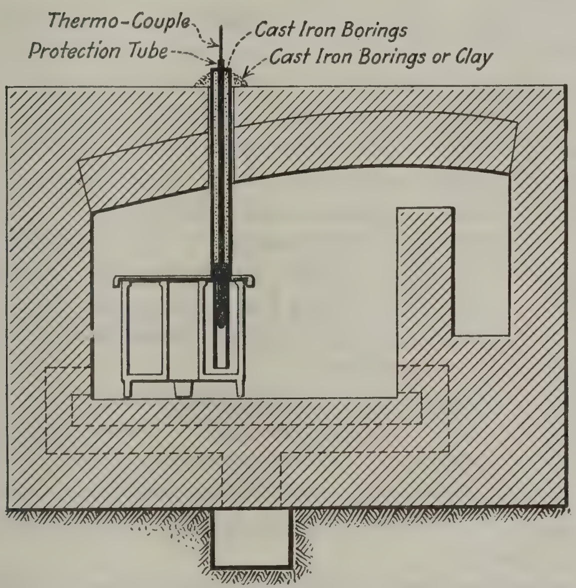

FIG. 1

good results. The use of molybdenum steel containing chromium is not recommended as this steel has an inherent tendency to distort in the carburizing operation.

5. Carburizing materials are divided into three general classes: solids, liquids, and gases, the solid type being generally used. The recommendations given apply to this type only. Commercial compounds are usually better than those made by the user. One part of new solid compound to three parts of used compound is recommended as a shop mixture. The used compounds should be screened to remove the dust. **Compounds.**

6. Packing pots are made from cast iron, cast and pressed steel and special heat-resisting alloys. The latter are recommended. Comparisons are usually made on a heat-hour-cost basis, the life of **Containers.**

the containers being determined by actual service and this figure divided into the cost of the pot which gives the cost per service-heat-hour. The proportions of the pot in relation to the parts to be carburized is of primary importance as well as the proper distribution of metal in the walls, reinforcement ribs, legs, etc. A compromise in size is found between the very small containers which are expensive in handling practice and the large containers which have a tendency to heat unevenly. The size of the pot should be governed by the size of the parts.

Temperature. 7. The effective temperature is the temperature inside the pot. The carburizing time should be designated as the number of hours at the carburizing temperature. The heating through of the pots is judged in two ways: by estimating with the eye, and by placing thermocouples inside the pot as shown in Fig. 1. The latter method is recommended except on pots which are so small that it would be impracticable to place a thermocouple inside them.

Pyrometers. 8. The use of recording pyrometers is recommended. Where the method shown in Fig. 1 is used, an extra couple should be placed in the furnace so that the furnace temperature may be more readily controlled, as otherwise excessive furnace temperatures or extremely variable heats may be obtained.

Heating. 9. Quick heating to the carburizing temperature will cause more distortion of the parts than slow heating. The temperature in any pot must be comparable with the temperature inside any other. To this end, the furnace must be designed to heat evenly and the pots must be spaced on the furnace hearth so that they will be heated evenly and uniformly.

Carburized Zone. 10. The carbon content at the surface of the work increases with the carburizing temperature. The formation of free carbide at the grain boundaries is undesirable, as it leaves a very brittle surface exposed to the service stresses. The surface carbon should not exceed 1.15 per cent. This will be reduced by the finish grinding operation. The carbon content is governed by the carburizing compound and the temperature while gradation depends upon the analysis of the steel and the rate of cooling. The allowable temperature depends upon the nature of the work. Temperatures of 941 to 954° C. (1725 to 1750° F.) may be used where speed and low expense are primary considerations. For general practice, 899° C. (1650° F.) is recommended. The carburizing temperature should be lowered about 38° C. (100° F.) when the regenerative quench is not to be used. Thin cases, of 0.010 to 0.015 in., are better obtained with a carburizing temperature of 843° C. (1550° F.).

TABLE I.—RECOMMENDED TEMPERATURES AND COOLING MEDIUMS TO BE USED FOR CARBURIZING AND HEAT TREATMENT OF CARBURIZED OBJECTS.

The temperatures and cooling mediums given in this table are dependent upon and must be varied according to the instructions given in the main section of the recommended practice. Particular attention should be given to varying the regenerative quench with the size of the part and the degree of distortion which must be avoided.

S. A. E. Steel No.	Composition						Preliminary Heat Treatments (use either A or B)									Carburizing			Refining and Hardening Treatments								
							Treatment A			Treatment B																
	Carbon, per cent	Manganese, per cent	Phosphorus, maximum per cent	Sulfur, maximum per cent	Nickel, per cent	Chromium, per cent	Normalizing Temperature		Cool in	Quenching Temperature		Cool in	Reheat to		Cool in	Temperature		Cool in	Regenerative Temperature		Cool in	Hardening Temperature		Cool in	Tempering Temperature	
							deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.		deg. Cent.	deg. Fahr.
1020	0.15–0.25	0.30–0.60	0.045	0.05	....	....	900	1650	air	900	1650	oil	595	1100	air	900	1650	pots	871	1600	oil	775	1425	brine	205	400
1015	0.10–0.20	0.30–0.60	0.045	0.05	....	....	900	1650	air	900	1650	or	595	1100	air	900	1650	pots	871	1600	oil	775	1425	brine	205	400
1025	0.20–0.30	0.50–0.80	0.045	0.05	....	....	900	1650	....	.....	.....	water	595	1100	air	900	1650	pots	871	1600	oil	775	1425	brine	205	400
2320	0.15–0.25	0.50–0.80	0.04	0.045	3.25–3.75	....	925	1700	air	830	1525	oil	595	1000	air	900	1650	pots	829	1525	oil	745	1375	oil	165	325
2315	0.10–0.20	0.30–0.60	0.04	0.045	3.25–3.75	....	.....	.....	....	.....	.....	.....	.....	.....	....	.....	.....	.....	.....	.....	....	.....	.....	.....	.....	.....
2512	max. 0.17	0.30–0.60	0.04	0.045	4.50–5.25	....	900	1650	air	830	1525	oil	595	1100	air	885	1625	pots	830	1525	oil	745	1375	oil	165	325
3115	0.10–0.20	0.30–0.60	0.04	0.045	1.00–1.50	0.45–0.75	925	1700	air	870	1600	oil	595	1000	air	900	1650	pots	845	1550	oil	760	1400	oil	165	325
3120	0.15–0.25	0.30–0.60	0.04	0.045	1.00–1.50	0.45–0.75	925	1700	air	870	1600	water	595	1100	air	900	1650	pots	845	1550	oil	{760 {782	1400 1440	water oil	165 165	325 325
3215	0.10–0.20	0.30–0.60	0.04	0.040	1.50–2.00	0.90–1.25	925	1700	air	845	1550	oil	620	1150	air	900	1650	pots	845	1550	oil	774	1425	oil	165	325
3220	0.15–0.25	0.30–0.60	0.04	0.040	1.50–2.00	0.90–1.25	925	1700	air	845	1550	oil	620	1150	air	900	1650	pots	845	1550	oil	760	1400	oil	175	350
3312	max. 0.17	0.30–0.60	0.04	0.040	3.25–3.75	1.25–1.75	900	1650	air	830	1525	oil	650	1200	air	885	1625	pots	855	1575	oil	760	1400	oil	190	375
3325	0.20–0.30	0.30–0.60	0.04	0.040	3.25–3.75	1.25–1.75	.....	.....	....	815	1500	oil	650	1200	air	885	1625	pots	855	1575	oil	760	1400	oil	190	375
3415	0.10–0.20	0.45–0.75	0.04	0.040	2.75–3.25	0.60–0.95	900	1650	air	845	1550	oil	620	1150	air	885	1625	pots	845	1550	oil	760	1400	oil	175	350
6120[a]	0.15–0.25	0.50–0.80	0.04	0.04	....	0.80–1.10	900	1650	air	900	1650	oil	595	1100	air	899	1650	pots	885	1625	oil	802	1475	water	190	375

[a] This steel is a chromium-vanadium steel, the vanadium requirement being 0.15 per cent minimum with a desired content of 0.18 per cent.

Measurement. 11. The "depth of case" shall be taken to be the depth of the refined case to be observed with the naked eye after carburizing, hardening at the temperature giving maximum case refinement, and breaking for fracture.

Regenerative Quench. 12. To obtain the best core condition, the part should be allowed to cool in the pots, outside the furnace, reheated and quenched from a temperature which will refine the core and break up any undesirable structures of cementite. This is called a regenerative quench.

TABLE II.—COMMON DEFECTS IN CARBURIZED OBJECTS.

Defect	Cause	Indication	Correction
Depth of case and carbon content outside specified limits.	A. Carburizing temperature not correct.		
	A1. Furnace temperature uneven. Temperature of containers not uniform.	Results vary in the same furnace and from the same containers.	Redesign furnaces. Modify heating practice. Allow longer time for bringing furnaces to temperature.
	A2. Faulty pyrometer readings.	Entire furnace charges unsatisfactory.	Check pyrometer system.
	A3. Carburizing compound too weak or too strong.	Results from entire department vary from usual results.	Check carburizing compound before accepting shipment. Change time and temperature until desired results are obtained.
Soft spots obtained in hardening.	B1. Improper hardening. Temperature low or uneven.	File test.	Raise hardening temperature.
	B2. Improper quench.	File test.	Use faster quench, brine or spray.
	B3. Surface decarburized.	File test. Determine whether decarburization occurred in carburizing or hardening furnace.	Correct carburizing practice. Correct heating practice for hardening.
	B4. Low carbon content or uneven penetration.	As above for B3.	Correct carburizing practice. Try other compound. Check pyrometers.
Unsatisfactory fractures.	C1. Too high hardening temperatures.	Fracture test.	Decrease or adjust hardening temperature.
	C2. High surface carbon (freckled edges).	Fracture test.	Decrease hardening temperature. Use double quench with rather high temperature for regenerative quench. Use weaker compound.

Cooling of Pots. 13. The cooling of the carburized parts should take place in the containers, outside of the furnace. Where cost of production is vital, the parts may be quenched directly from the pot in either oil or water.

Grinding. 14. In determining the depth of case desired, sufficient allowance must be made for the metal which will later be removed by grinding. A tempering operation will tend to eliminate the tendency for grinding cracks to form.

Hardening. 15. The hardening temperature of the case is below the critical temperature of the core and the grain size produced in the core, by the carburizing temperature or regenerative quench, is not affected. The hardening temperature should be chosen as the temperature which gives the best fracture on several dummies or experimental pieces hardened from various temperatures. The figures given in Table I are the middle of the probable range of hardening temperatures.

16. The tendency to distort may be avoided by a preliminary heat treatment, either before or after the rough machining operation. The method of carburizing must be studied and the subsequent hardening operations carried on with extreme care as to the heating and cooling cycles. In many cases, the regenerative quench must be omitted as the high temperature of this operation causes too much warping. When omitted, the carburizing temperature should be lowered about 38° C. (100° F.). **Distortion.**

17. Tempering may be carried on at low temperatures, for the relief of strains, or at high temperatures, for the increase of toughness and uniformity without materially decreasing the hardness. An oil bath is preferable to an open furnace for temperatures up to 316° C. (600° F.). For simple carbon steels, a softening of the surface is found when the tempering temperature exceeds 190 to 204° C. (375 to 400° F.). For maximum hardness the temperature should be kept below this figure. **Tempering.**

18. Table II shows in condensed form the most common defects in carburized work together with the possible causes and methods for their identification and correction.

TENTATIVE SPECIFICATIONS

FOR

PHOSPHOR TIN[1]

Serial Designation: B 51 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

1. These specifications cover phosphor tin in one grade.

I. MANUFACTURE

Material Covered. 2. The manufacturer shall use new tin of uniform quality throughout in the manufacture of phosphor tin.

3. The manufacturer or vendor shall furnish phosphor tin ingots in the form of notched slabs of a size that can be handled conveniently.

4. Phosphor tin shall be clean, free from defects, and uniform in quality.

II. CHEMICAL PROPERTIES AND TESTS

(*A*) *Chemical Composition*

Chemical Composition. 5. The material shall conform to the following requirements as to chemical composition:

Phosphorus, minimum	3.50 per cent
Phosphorus plus tin, minimum	99.50 "

(*B*) *Sampling and Testing*

Sampling. 6. One ingot from each melt or portion of melt shall be selected and not less than 140 g. of clean fine drillings or sawdust shall be taken from the ingot and mixed thoroughly. The ingots selected shall be drilled completely through or one-half through from top to bottom, the surface material being rejected.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

III. MARKING

7. A brand and melt number shall be cast or otherwise plainly marked upon each slab or ingot by which the manufacturer and heat may be identified. Marking may be done either by stamping or painting. **Markings.**

IV. CLAIMS

8. Claims to be considered shall be made in writing within ten days of receipt of material at the purchaser's plant and the results of the purchaser's tests shall be given. The vendor shall within ten days of such claim, either agree to satisfy the claim or send a representative to the purchaser's plant to resample the shipment as specified in Section 6. Samples so taken shall be sealed and submitted to a mutually agreeable umpire, whose determination shall be final. **Claims.**

9. The expenses of the umpire analysis shall be paid by the loser, or divided in proportion to the concession made, in case of compromise. In case rejection is established, the damage shall be limited to the payment of freight both ways by the vendor for the substitution of an equivalent weight of metal meeting specifications.

TENTATIVE SPECIFICATIONS

FOR

PHOSPHOR COPPER[1]

Serial Designation: B 52 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting **criticism** and suggestions. It is not a Standard of the Society and is subject to annual **revision.**

ISSUED, 1924

1. Under these specifications phosphor copper is considered in two grades.

I. MANUFACTURE

2. The manufacturer shall use new metal, or new metal and scrap resulting from the manufacture of ingots of similar composition, in the manufacture of this material.

3. The manufacturer or vendor shall furnish phosphor copper as ingots in the form of notched slabs, weighing from 15 to 20 lb., unless otherwise specified in the contract.

4. Phosphor copper shall be clean, free from defects and uniform in quality.

II. CHEMICAL PROPERTIES AND TESTS

(A) Chemical Composition

5. The material shall conform to the following requirements as to chemical composition:

	Phosphorus, Minimum, per cent	Phosphorus Plus Copper, Minimum, per cent	Iron, Maximum, per cent
Grade A	14.0	99.75	0.15
Grade B	10.0	99.75	0 15

(B) Sampling and Testing

6. One ingot from each melt or portion of melt shall be selected and not less than 140 g. of clean fine drillings or sawdust shall be taken from the ingot and mixed thoroughly. The ingots selected

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

shall be drilled completely through or one-half through from top and bottom, the surface material being rejected.

III. MARKING

7. A brand and melt number shall be cast or otherwise plainly marked upon each slab or ingot by which the manufacturer and heat may be identified. Marking may be done either by stamping or painting.

IV. CLAIMS

8. Claims to be considered shall be made in writing within ten days of receipt of material at the purchaser's plant, and the results of the purchaser's tests shall be given. The vendor shall within ten days of such claim, either agree to satisfy the claim or send a representative to the purchaser's plant to resample the shipment as specified in Section 6. Samples so taken shall be sealed and submitted to a mutually agreeable umpire, whose determination shall be final.

9. The expenses of the umpire analysis shall be paid by the loser, or divided in proportion to the concession made, in case of compromise. In case rejection is established, the damage shall be limited to the payment of freight both ways by the vendor for the substitution of an equivalent weight of metal meeting specifications.

TENTATIVE SPECIFICATIONS

FOR

SILICON COPPER[1]

Serial Designation: B 53 – 24 T

This is a Tentative Standard only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

1. These specifications cover silicon copper in one grade.

I. MANUFACTURE

2. The manufacturer shall use new metal, or new metal and scrap resulting from the manufacture of ingots of similar composition in the manufacture of this material.

3. The manufacturer or vendor shall furnish silicon copper in the form of ingots or notched slabs of such size that they can be handled conveniently.

4. Silicon copper shall be clean, free from defects, and uniform in quality.

II. CHEMICAL PROPERTIES AND TESTS

(A) Chemical Composition

5. The material shall conform to the following requirements as to chemical composition:

Silicon	10 to 12 per cent
Tin, maximum	0.25 "
Zinc, maximum	0.25 "
Iron, maximum	0.75 "
Aluminum, maximum	0.25 "
Copper	Remainder

The sum of the copper, silicon and iron content shall be not less than 99.4 per cent.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

(*B*) *Sampling and Testing*

6. One ingot from each melt or portion of melt shall be selected and not less than 140 g. of clean fine drillings or sawdust shall be taken from the ingot and mixed thoroughly. The ingots selected shall be drilled completely through or one-half through from top and bottom, the surface material being rejected.

III. MARKING

7. A brand and melt number shall be cast or otherwise plainly marked upon each slab or ingot by which the manufacturer and heat may be identified. Marking may be done either by stamping or painting.

IV. CLAIMS

8. Claims to be considered shall be made in writing within ten days of receipt of material at the purchaser's plant, and the results of the purchaser's tests shall be given. The vendor shall within ten days of such claim, either agree to satisfy the claim or send a representative to the purchaser's plant to resample the shipment as specified in Section 6. Samples so taken shall be sealed and submitted to a mutually agreeable umpire, whose determination shall be final.

9. The expenses of the umpire analysis shall be paid by the loser, or divided in proportion to the concession made, in case of compromise. In case rejection is established, the damage shall be limited to the payment of freight both ways by the vendor for the substitution of an equivalent weight of metal meeting specifications.

TENTATIVE SPECIFICATIONS

FOR

ALUMINUM BASE ALLOY SAND CASTINGS[1]

Serial Designation: B 26 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

Material Covered. 1. These specifications cover commercial aluminum base alloy sand castings having a specific gravity of 3 and less.

I. MANUFACTURE

Process. 2. The alloy may be made by any approved method.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 3. The following alloys are described by these specifications; they shall conform to the following requirements as to chemical composition:

Alloy	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Magnesium, per cent	Manganese, per cent	Zinc, per cent	Remarks
A.....	not under 96.5	1 to 1.5	not over 0.5	not over 0.5	trace	0.7 to 2.0	trace	
B.....	not under 92.5	not over 0.6	not over 1.0	4.5 to 6.0	trace	not over 0.2	not over 0.20	
C.....	not under 90.0	7 to 8.5	......	......	......	......	not over 0.20	Total of Fe, Si, Mn, Zn and Sn not to exceed 1.70 per cent.
D....	88 to 92	6 to 8.0	not over 1.50	......	......	......	not over 2.50	Total of Si, Mn, and Sn not to exceed 1.00 per cent.

Sampling. 4. (*a*) The sample for chemical analysis may be taken either by sawing, drilling or milling the casting or tension test specimen and shall represent the average cross-section of the piece.

(*b*) The saw, drill, cutter or other tool used shall be thoroughly cleaned. No lubricant shall be used in the operation, and the sawdust

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

These Tentative Specifications, when adopted as standard, will supersede the present Standard Specifications for Light Aluminum Casting Alloys (Serial Designation: B 26 – 21), 1924 Book of A.S.T.M. Standards.

or metal chips shall be carefully treated with a magnet to remove any particles of iron introduced in taking the sample.

III. PHYSICAL PROPERTIES AND TESTS

5. The alloys shall conform to the following minimum requirements as to tensile properties: **Tension Tests.**

Alloy	Minimum Tensile Strength, lb. per sq. in.	Minimum Elongation in 2 in., per cent
A	18 000	8
B	16 000	3.5
C	18 000	..
D	19 000	..

6. (*a*) Two test bars shall be an integral part of large castings, or cast separately in the case of small castings to represent a lot or melt, and shall be molded in a manner similar to the castings which they represent. If the castings are heat treated, the test bars representing such castings shall be similarly heat treated. **Test Specimens.**

(*b*) The manufacturer and purchaser shall agree whether the test bars can be attached to castings, on the locations of the bars, on the castings to which bars are to be attached, on the method of casting unattached bars and whether a "cast-to-size bar" or a "machined-to-size bar" shall be used.

(*c*) The "machined-to-size bar" shall be cast according to the dimensions shown in Fig. 1, and machined to conform to the dimensions shown in Fig. 2. The "cast to size" bar shall be cast according to the dimensions shown in Fig. 2 and shall be tested without machining. The ends shall be of a form to fit the holders of the testing machine in such a way that the load shall be axial.

7. (*a*) Tests shall as far as possible be made by heats or melts, but unless otherwise agreed, two tension tests shall be made upon each unit lot of 500 lb. or single delivery of less than 500 lb. **Number of Tests.**

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded; in which case the manufacturer and the purchaser or his representative shall agree upon the selection of another specimen in its stead.

IV. INSPECTION AND REJECTION

8. (*a*) Inspection may be made at the manufacturer's works where the castings are made, or at the point at which they are received, at the option of the purchaser. **Inspection.**

(*b*) If the purchaser elects to have inspection made at the manufacturer's works, the inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to

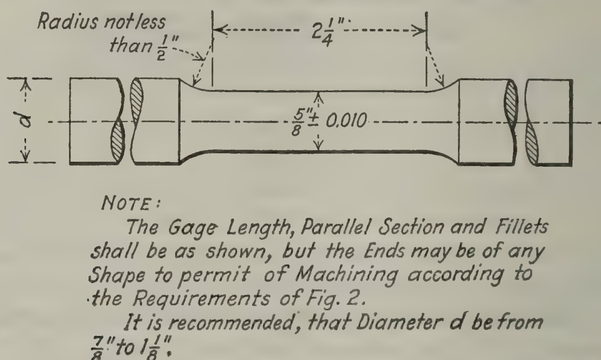

FIG. 1

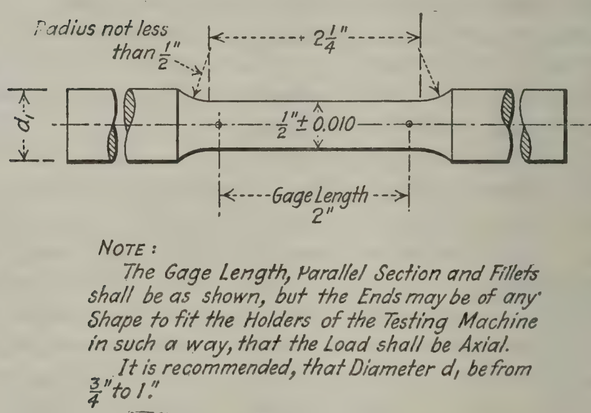

FIG. 2

satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 9. Castings which show injurious defects revealed by machining operations subsequent to acceptance may be rejected, and if rejected, shall be replaced by the manufacturer free of charge to the purchaser. The full weight of the original material rejected shall be returned to the manufacturer.

Explanatory Notes

The Naval Gun Factory has been the largest user of Alloy A. Its resistance to salt-water corrosion makes it suitable for marine equipment. Although very large castings have been successfully poured from it, Alloy A is generally regarded as difficult to handle in the foundry. Its proportional limit is rather low.

Alloy B possesses very good foundry characteristics; the melt is quite fluid and the solidification shrinkage is low. It is especially suitable for the production of dense non-porous castings and of intricate shapes containing both heavy and thin sections. This alloy is the most resistant to corrosion of all the commonly used aluminum casting alloys. It is somewhat more difficult to machine than the other alloys and, as in the case of Alloy A, possesses a rather low proportional limit.

Alloys C and D are similar, both being substantially aluminum-copper alloys. The composition limits are such that secondary metal can be used in the manufacture of Alloy D. While no lower limits are placed on the iron and zinc in Alloy D, it is assumed they will be present and they are stated to improve the machining qualities of the castings.

The pattern maker's shrinkage is usually taken as 0.156 in. per foot for all these alloys. The weight lies within the range of 0.096 lb. per cu. in. for Alloy B, and 0.103 lb. per cu. in. for Alloy C, corresponding to the specific gravities 2.66 and 2.85, respectively. These are approximate values, since the weights will vary with the actual compositions of the individual samples.

Section 5.—The elongations of Alloys C and D are usually so low as to present difficulties in their accurate measurement. The fact that a very large percentage of all aluminum castings produced in this country is made from these alloys, is evidence that the low elongation is not indicative of excessive brittleness.

Section 6.—Some latitude has been allowed in the matter of test specimens; test bars "cast to size" are stronger than those which are "machined to size" and the tensile properties specified are regularly obtained in commercial production only with the "cast to size" bar. When "machined to size" specimens are required to meet the values given in these specifications, rejections may be expected to run higher than those experienced in usual commercial production. Owing to insufficient information being available on the tensile properties of "machined to size" specimens it was not possible to include a separate table of values for this type of specimen.

It is strongly recommended that a self-centering form of tension test specimen holder be used. Most aluminum alloys are brittle and give lower and less uniform test results when tested in ordinary wedge grips without centering.

TENTATIVE SPECIFICATIONS

FOR

BRONZE TROLLEY WIRE[1]

Serial Designation: B 9 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

I. MANUFACTURE

Scope. 1. These specifications cover round and grooved bronze trolley wire.

Material. 2. The material used shall be bronze of such nature and composition as to secure by proper treatment the qualities hereinafter specified for the finished wire. The copper used shall be electrolytic or low-resistance lake copper conforming in quality and purity to the requirements of either the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (Serial Designation: B 5) or of the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (Serial Designation: B 4) of the American Society for Testing Materials.[2]

Bronze of two qualities may be required:

(*a*) High Strength.

(*b*) Medium Strength.

II. PHYSICAL PROPERTIES AND TESTS

(*A*) *Round Wire.*

Tensile Strength and Elongation. 3. (*a*) Round wire shall be so drawn that its tensile strength and elongation shall not be less than the values given in Table I.

[1] These specifications were prepared by Committee B-1 on Copper Wire in cooperation with a committee of the American Electric Railway Engineering Association. The specifications of the two organizations are identical save with respect to the requirements for the twist test, Section 4. Further cooperative work with respect to these requirements is being undertaken.

Criticisms of these Tentative Specifications are solicited and should be directed to Mr. J. A. Capp, Chairman of Committee B-1 on Copper Wire, General Electric Co., Schenectady, N. Y.

These tentative specifications, when adopted as standard, will supersede the present Standard Specifications for High-Strength Bronze Trolley Wire, Round and Grooved (Serial Designation: B 9 – 16), 1924 Book of A.S.T.M. Standards.

[2] 1924 Book of A.S.T.M. Standards.

The elongation shall be determined as the permanent increase in length due to the breaking of the wire in tension measured between bench marks placed upon the wire originally 10 in. apart. The fracture shall be between the bench marks and not closer than 1 in. to either bench mark.

(*b*) Tests upon a section of wire containing a braze shall show at least 95 per cent of the tensile strength of the unbrazed wire.

Elongation tests shall not be made on test sections including brazes.

Twist Test. 4. For the purpose of determining and developing defects which may be prejudicial to the life of trolley wire, owing to its peculiar service, as compared to that of copper wire for other purposes, round wire shall be subjected to the following twisting test. Three twist tests shall be made upon samples 10 in. in length between the holders

TABLE I.—VALUES FOR TENSILE STRENGTH AND ELONGATION OF ROUND WIRE.

Diameter, in.	Area, circular mils	High-Strength Bronze Wire: Tensile Strength, lb. per sq. in.	High-Strength Bronze Wire: Elongation in 10 in., per cent	Medium-Strength Bronze Wire: Tensile Strength, lb. per sq. in.	Medium-Strength Bronze Wire: Elongation in 10 in., per cent
0.548.....	300 000	70 000	4.50	60 000	4.50
0.460.....	211 600	73 000	3.75	62 000	3.75
0.410.....	168 100	76 000	3.25	64 000	3.25
0.365.....	133 225	79 000	2.80	66 000	2.80
0.325.....	105 625	81 000	2.40	69 000	2.40

of the machine. The twisting machine shall be so constructed that there is a linear motion of the tail stock with respect to the head. The twist shall be applied not faster than 10 turns per minute. All three samples shall be twisted to destruction and shall not reveal under test any seams, pits, slivers, or surface imperfections of sufficient magnitude to indicate inherent defects or imperfections in the wire. At the time of fracture the wire shall be twisting with reasonable uniformity. Wire shall not be considered satisfactory which does not withstand at least twenty turns before breaking.

Resistivity. 5. Electric resistivity shall be determined upon fair samples by resistance measurements at a temperature of 20° C. (68° F.).

High-strength wire shall not exceed in resistivity 0.38320 ohms per meter gram. Medium-strength wire shall not exceed in resistivity 0.23574 ohms per meter gram.

The values given above are based on bronzes of copper-tin compositions and represent classes of such wire formerly referred to as 40-per-cent and 65-per-cent conductivity wire.

Lower resistivities in wires meeting the physical qualifications of these specifications may be obtained by the use of other alloys.

(*B*) *Grooved Wire.*

Physical Properties and Tests. 6. (*a*) The physical tests for grooved wire shall be made in the same manner as those upon the round wire. The tensile strength of grooved wire shall be at least 95 per cent of that required for round wire of the same nominal cross-sectional area; the elongation shall be

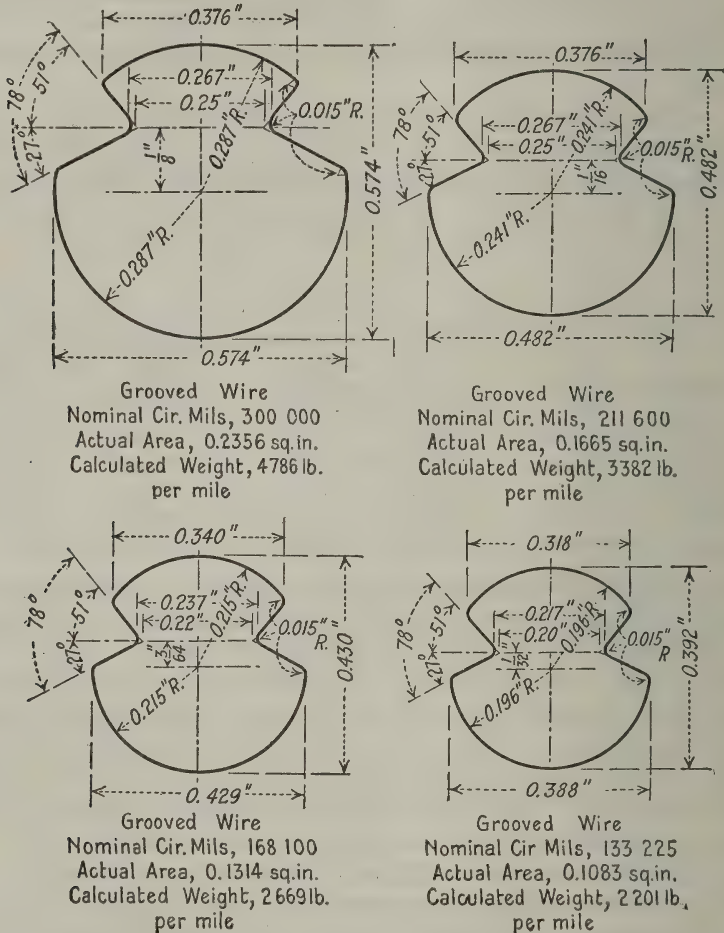

FIG. 1.

the same as that required for round wire of the same nominal cross-sectional area. The twist test shall be omitted.

(*b*) Tests upon a section of wire containing a braze shall show at least 95 per cent of the tensile strength of the unbrazed wire.

Elongation tests shall not be made on test sections including brazes.

7. The requirements for resistivity of grooved wire shall be the same as those for round wire of the same nominal cross-sectional area. **Resistivity.**

III. DIMENSIONS AND PERMISSIBLE VARIATIONS

(A) Round Wire.

8. (*a*) The size shall be expressed as the diameter of the wire in decimal fractions of an inch, using not more than three places of decimals, *i. e.*, in mils. **Dimensions and Permissible Variations.**

(*b*) Wire shall be truly cyclindrical in form. The diameter shall not vary more than 1 per cent over or under the diameter specified.

(B) Grooved Wire.

9. Standard sections of grooved trolley wire shall be those known as the "American Standard Grooved Trolley Wire Sections" (the Standard Design of the American Electric Railway Engineering Association), shown in Fig. 1. **Sections.**

10. (*a*) Size shall be expressed as the nominal area of cross-section in circular mils, the standard sizes being as follows: **Dimensions and Permissible Variations.**

Nominal Area, circular mils	Actual Area, sq. in.	Calculated Weight Based on Actual Area, lb. per mile
300 000	0.2356	4786
211 600	0.1665	3382
168 100	0.1314	2669
133 225	0.1083	2201

(*b*) The weight of grooved trolley wire shall not vary more than 4 per cent over or under the weights specified in Paragraph (*a*).

IV. FINISH

11. (*a*) The wire shall be of uniform size, shape, and quality throughout, and shall be free from all scale, flaws, splits and scratches not consistent with the best commercial practice. **Finish.**

(*b*) Necessary brazes in trolley wire shall be made in accordance with the best commercial practice.

V. PACKING

12. All wire shall be shipped on substantial reels, suitable for the weight of the wire handled, and shall be well protected from injury. If reels are to be lagged, it shall be so specified by the purchaser. **Packing and Shipping.**

The ends shall be securely fastened to the sides of the reels with no less than six staples. The staples shall be at least 2 in. in length

and made from not less than 0.145-in. diameter wire. Care shall be exercised in stapling not to damage the surface of the exposed layer of wire. All reels shall have the arbor holes reinforced with steel plates. Reels shall be marked legibly and indelibly with a serial number, size, kind, length and weight of wire, and such other information as is desired by the purchaser. The length or weight of wire to be wound upon reels shall be agreed upon in placing individual orders.

VI. INSPECTION AND REJECTION

Inspection. 13. All tests governing the acceptance or rejection of the wire, unless otherwise specified, shall be made at the place of manufacture with apparatus furnished by the manufacturer and in the presence of the purchaser, or his representative, who shall be furnished a copy of the tests. Every facility shall be given the purchaser, or his representative, to satisfy him as to the reliability of the results before the wire is delivered. If the purchaser waives inspection, and if he so elects at that time, he shall be furnished with a certified copy of tests made by the manufacturer.

Rejection. 14. Any reel of wire which fails to meet the requirements hereinbefore specified may be rejected. Failure of 30 per cent of the number of reels ready for inspection at one time shall be deemed sufficient cause for the rejection of the whole lot.

TENTATIVE SPECIFICATIONS
FOR
SOFT RECTANGULAR COPPER WIRE[1]

Serial Designation: B 48 – 23 T

This is a Tentative Standard only, published for the purpose of eliciting criticism **and suggestions.** It is not a Standard of the Society and is subject to revision.

Issued, 1923.

I. MANUFACTURE

1. These specifications cover soft or annealed rectangular copper wire with rounded corners. **Scope.**

2. The copper shall conform in quality and purity to the requirements of either the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 4) or of the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 5) of the American Society for Testing Materials.[2] **Material.**

II. PHYSICAL PROPERTIES AND TESTS

3. The wire shall conform to the following requirements as to tensile properties: **Physical Tests.**

Thickness, in.	Maximum Tensile Strength, lb. per sq. in.	Minimum Elongation in 10 in., per cent
0 003 to 0.010	......	14
0.011 to 0.020	40 000	20
0.021 to 0.050	38 500	25
0.051 to 0.289	37 000	30
0.290 to 0.500	36 000	33

The elongation shall be measured between bench marks placed upon the wire originally 10 in. apart and the fracture shall be between the bench marks and not closer than 1 in. to either bench mark. If upon testing a sample from any coil or reel, the results do not conform to the specified requirements two additional samples shall be tested and the average of the three tests shall determine acceptance or rejection of the coil or reel.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. J. A. Capp, Chairman of Committee B-1 on Copper Wire, General Electric Co., Schenectady, N. Y.

[2] 1924 Book of A.S.T.M. Standards.

Electric Resistivity. 4. Electric resistivity (Note 1) shall be determined upon fair samples by resistance measurements at a temperature of 20° C. (68° F.) and shall not exceed 891.58 lb. per mile-ohm.

III. DIMENSIONS AND PERMISSIBLE VARIATIONS

Dimensions and Permissible Variations. 5. (*a*) The permissible variation in thickness, above or below the specified thickness, for different widths shall not exceed the limits given in the following table (Note 2):

Specified Thickness, in.	Permissible Variations		
	½ in. or Less in Width	Over ½ in. to 1 in. in Width	Over 1 In. in Width
0.050 or less	0.001 in.	0.001 in.	0.0015 in.
0.051 to 0.100	0.001 in.	0.001 in.	0.002 in.
0.101 to 0.200	1 per cent	1 per cent	0.0025 in.
0.201 to 0.300	1 per cent	1 per cent	0.003 in.
0.301 to 0.500	0.003 in.	1 per cent	1 per cent
0.501 or over	....	1 per cent	1 per cent

(*b*) A variation of ±0.003 in. will be permitted in the width of wire 0.300 in. or less in width; wire 0.301 to 1.50 in. in width may vary ±1 per cent; wire greater than 1.50 in. in width shall not vary more than ±0.016 in. from the specified width.

(*c*) Dimensions shall be expressed in decimals of an inch.

(*d*) Rectangular wire shall have edges with corners rounded to a radius as given in the following table:

Thickness, in.	Width, 0 to 0.438 in.	Width, 0.439 to 0.625 in.	Width, 0.626 in. or Over
0.050 or less	Half Round	Half Round	Half Round
0.051 to 0.105	0.025	Half Round	Half Round
0.106 to 0.125	0.031	Half Round	Half Round
0.126 to 0.165	0.031	0.047	0.063
0.166 to 0.225	0.047	0.047	0.063
0.226 to 0.438	0.063	0.063	0.094
0.439 to 0.688	.....	0.094	0.125
0.689 and above	.....	.....	0.188

Each coil shall be gaged at three places, one near each end and one approximately at the middle. From reels, approximately 12 ft. shall be reeled off; the wire shall be gaged in six places between the second and twelfth foot from the end. A coil or reel may be rejected if the average of the measurements obtained is not within the limits specified in Paragraphs (*a*) or (*b*).

IV. FINISH

Finish. 6. (*a*) The wire shall be free from all imperfections not consistent with the best commercial practice.

(*b*) Necessary brazes shall be made in accordance with the best commercial practice.

(*c*) The copper shall be thoroughly annealed after the last drawing or rolling.

V. PACKING

7. (*a*) Rectangular wire shall be shipped in coils or on reels as agreed upon by the purchaser and the manufacturer (Note 3). **Packages.**

(*b*) The wire shall be packed so as to be properly protected against injury during shipment.

(*c*) Unless otherwise agreed upon, the wire shall be shipped in continuous lengths as follows:

Thickness, in.	Width, in.	Minimum Weight per Length, lb.
Less than 0.025	0.500 or less	5
Less than 0.010	Over 0.500	5
0.010 to 0.019, inclusive	Over 0.500	10
0.025 to 0.034, "	0.500 or less	15
0.020 to 0.094, "	Over 0.500 to 0.750	25
0.020 to 0.094, "	Over 0.750 to 1.00	35
0.020 to 0.094, "	Over 1.00	50
0.035 to 0.094, "	0.500 or less	20
0.095 to 0.300, "	0.500 or less	40
0.301 to 0.500, "	0.500 or less	100
Over 0 094	Over 0.500	100

8. For the purpose of calculating weights, cross-sections, etc., the specific gravity of the copper shall be taken as 8.89 at 20° C. (68° F.) (Note 4). **Specific Gravity.**

VI. INSPECTION

9. Test and inspection shall be made at the place of manufacture. The manufacturer shall afford the inspector representing the purchaser all reasonable facilities to satisfy him that the material conforms to the requirements of these specifications. **Inspection.**

EXPLANATORY NOTES

Soft or annealed copper wire is wire which has been drawn and rolled by customary operations and annealed, and finished by cleaning when necessary to remove scale or oxide. The wire is so soft and ductile that it is easily marred and even stretched by careless handling, hence the necessity for confining specifications and inspection to wire in packages as it leaves the manufacturer, and before being put through processes incident to its use by the purchaser.

NOTE 1.—During the year 1913 the following resistivity was agreed upon internationally as standard for annealed copper:

0.15328 ohm per meter-gram at 20° C.

This resistivity is known as the International Annealed Copper Standard[1] and is equivalent to:

875.20 ohms per mile-pound at 20° C.
0.017241 ohm per meter-millimeter squared at 20° C.
1.7241 microhms per centimeter cubed at 20° C.
0.67879 microhms per inch cubed at 20° C.
10.371 ohms per mil-foot at 20° C.

For reducing the resistance of annealed copper from one temperature to another the following temperature coefficients should be used:

At 0° C., 0.00427; at 15° C., 0.00401; at 20° C., 0.00393; at 25° C., 0.00385.

In case it is desired to express the resistivity of copper in terms of conductivity, the International Annealed Copper Standard at 20° C. should be taken as 100-per-cent conductivity, and in reporting conductivity values this standard should be mentioned. It has been deemed preferable to express the requirements in standard specifications in the terms of quantities directly measurable, rather than by reference to some quantity whose standard value is the subject of agreement only. The use of the arbitrary term "conductivity" has no more warrant than the employment of arbitrary gage numbers. Therefore, in these specifications the requirements are stated as the maximum rejection limits to the resistivity.

For the convenience of those who are accustomed to express resistivity in any one of the several more or less common units, the following table of equivalents has been prepared, giving the resistivity of copper at 20° C.

887.05 lb. per mile-ohm is equal to:
0.15535 ohms per meter-gram,
1.7475 microhms per centimeter-cube,
0.68799 microhms per inch-cube,
10.512 ohms per mil-foot.

891.58 lb. per mile-ohm is equal to:
0.15614 ohms per meter-gram,
1.7564 microhms per centimeter-cube,
0.69150 microhms per inch-cube,
10.565 ohms per mil-foot.

NOTE 2.—The use of arbitrary gage numbers to express dimensions cannot be too strongly condemned. There are many such gages in existence, and confusion is to be expected unless the particular gage to be used is specified. Many of the gages have their dimensions stated in absurd figures, such as 0.090742 in., when it is not practical to measure dimensions in the fourth decimal place by workshop tools. Definite dimensions in measurable units are evidently preferable.

NOTE 3.—Attention is called to the necessity for the purchaser and manufacturer agreeing on the package weights which will be standard under any individual contract.

NOTE 4.—The specific gravity of copper was formerly standardized in A.S.T.M. specifications at 8.90. The value has been changed to 8.89, since that is the value adopted as standard by the American Institute of Electrical Engineers and the International Electro-Technical Commission.

[1] See U. S. Bureau of Standards *Circular No. 31* for further details.

TENTATIVE SPECIFICATIONS

FOR

HOT-ROLLED COPPER RODS FOR WIRE DRAWING[1]

Serial Designation: B 49 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923

I. MANUFACTURE

Scope. 1. These specifications cover black hot-rolled copper rods for wire-drawing purposes.

Material. 2. The copper shall conform in quality and purity to the requirements of either the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 4) or of the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 5) of the American Society for Testing Materials.[2]

3. (*a*) The rod shall be hot rolled from standard wire bars.

(*b*) The rods shall be furnished in one continuous length without joints.

II. PHYSICAL PROPERTIES AND TESTS

Physical Tests. 4. (*a*) The rods shall conform to the following requirements as to tensile properties:

The elongation in 10 in. shall not be less than 25 per cent. The elongation shall be measured between bench marks placed upon the wire originally 10 in. apart and the fracture shall be between the bench marks and not closer than 1 in. to either bench mark. If upon testing a sample from any coil or reel the results do not conform to the specified requirements, two additional samples shall be tested and the average of the three tests shall determine acceptance or rejection of the coil or reel.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. J. A. Capp, Chairman of Committee B-1 on Copper Wire, General Electric Co., Schenectady, N. Y.

[2] 1924 Book of A.S.T.M. Standards.

(*b*) Two tests shall be made from each carload of 50,000 lb. or less.

Electric Resistivity. 5. Electric resistivity shall be determined upon fair samples by resistance measurements at a temperature of 20° C. (68° F.) and shall not exceed 891.58 lb. per mile-ohm.

III. DIMENSIONS AND PERMISSIBLE VARIATIONS

Dimensions and Permissible Variations. 6. (*a*) The size shall be expressed as the diameter of the rod in decimal fractions of an inch.

(*b*) The rods shall be hot rolled to nominal size within the following permissible variations:

Diameter, in.	Permissible Variations, in.	Diameter, in.	Permissible Variations, in.
0.250	+0.020	0.875	±0.020
0.3125	±0.010	1.00	±0.020
0.375	±0.010	1.125	±0.020
0.500	±0.015	1.250	±0.020
0.625	±0.015	1.375	±0.030
0.750	±0.015		

Sizes between those listed shall take the variation for the next larger size in the table.

Specific Gravity. 7. For the purpose of calculating weights, cross-sections, etc., the specific gravity of the copper shall be taken as 8.89 at 20° C (68° F.).

IV. FINISH

Finish. 8. The rod shall be free from all imperfections not consistent with the best commercial practice.

V. PACKING

Packages. 9. Unless otherwise agreed upon, the rods shall be shipped in coils approximately 24 in. in inside diameter and each coil shall be bound tightly together with copper wire at two points approximately opposite to each other.

VI. INSPECTION

Inspection. 10. The tests and inspection shall be made at the place of manufacture. The manufacturer shall afford the inspector representing the purchaser all reasonable facilities to assure him that the material conforms to all the requirements of the specifications.

TENTATIVE SPECIFICATIONS
FOR
MANGANESE-BRONZE INGOTS FOR SAND CASTINGS[1]

Serial Designation: B 7 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

1. These specifications cover the copper-zinc alloy known commercially as manganese bronze. **Material Covered.**

I. MANUFACTURE

2. The bronze shall be cast in the form of ingots having notched flat bottoms, approximately 3 by $2\frac{3}{4}$ in. wide by 12 in. long, properly tapered to strip easily from an iron mold. **Size of Ingots.**

II. CHEMICAL PROPERTIES[2]

3. The bronze shall conform to the following requirements as to chemical composition: **Chemical Composition.**

	Maximum	Minimum
Copper, per cent	60	55
Zinc, per cent	42	38
Tin, per cent	1.5	0.0
Manganese, per cent	3.5	0.0
Aluminum, per cent	1.5	0.0
Iron, per cent	2.	0.0
Lead, per cent	0.2	0.0

III. PHYSICAL PROPERTIES AND TESTS

4. The bronze shall conform to the following minimum requirements as to tensile properties: **Tension Tests.**

Tensile strength	70,000 lb. per sq. in.
Elongation in 2 in.	30 per cent

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

These Tentative Specifications are in effect a revision of the Standard Specifications for Manganese-Bronze Ingots for Sand Castings. The Standard Specifications, which were last published under the Serial Designation: B 7 – 14, have accordingly been withdrawn.

[2] Because of claims made of the improved qualities of magnanese bronze made from virgin metal (copper conforming to the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 5) and zinc conforming to the requirements of Grade 2 or better of the Standard Specifications for Spelter (Serial Designation: B 6) of the American Society for Testing Materials, 1924 Book of A.S.T.M. Standards) the buyer may, at his option, elect when contracting that these materials only be used.

Tension Test Specimens. 5. (*a*) The tension test specimen shall be cut from one corner near the bottom of the test ingot.

(*b*) The specimen shall conform to the dimensions shown in Fig. 1.

Number of Tests. 6. (*a*) One test ingot shall be selected by the inspector to represent 10,000 lb. of ingots or fraction thereof.

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded, in which case two additional test specimens may be selected by the inspector from the same lot, and tested to represent the lot.

IV. MARKING

Marking. 7. Each ingot shall be stamped with its proper heat or charge number.

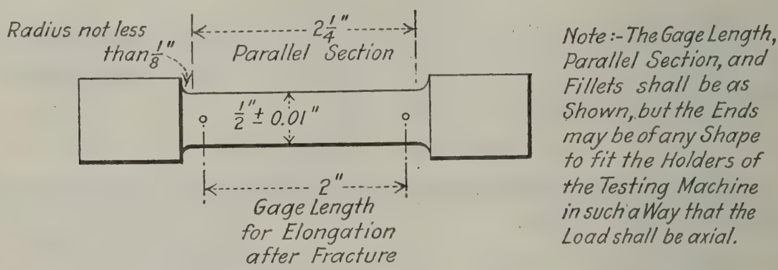

FIG. 1

V. INSPECTION AND REJECTION

Inspection. 8. (*a*) Inspection may be made at the manufacturer's works where the castings are made or at the point at which they are received, at the option of the purchaser.

(*b*) If the purchaser elects to have inspection made at the manufacturer's works, the inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 9. (*a*) If the test ingots selected to represent a lot fail to conform to the requirements specified in Sections 3 or 4, all ingots in such lot will be rejected, irrespective of the heat or heats from which the test ingots are selected.

TENTATIVE SPECIFICATIONS

FOR

MANGANESE-BRONZE SAND CASTINGS[1]

Serial Designation: B 54 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

Material Covered.

1. These specifications cover castings made from the copper-zinc alloy known commercially as manganese bronze.

I. MANUFACTURE

Material Used.

2. Material used in these castings shall conform to the requirements of the Tentative Specifications for Manganese-Bronze Ingots for Sand Castings (Serial Designation: B 7 – 24 T) of the American Society for Testing Materials.[2] At least 35 per cent of the mixture shall consist of ingots manufactured in accordance with the Tentative Specifications for Manganese-Bronze Ingots and the remainder shall consist of risers, sprues, and defective castings, accruing from the manufacture of castings in accordance with these specifications. Zinc additions shall be of zinc conforming to the requirements of Grade 2 or better of the Standard Specifications for Spelter (Serial Designation: B 6) of the American Society for Testing Materials.[3]

II. CHEMICAL PROPERTIES

Chemical Composition.

3. The bronze shall conform to the following requirements as to chemical composition:

	Maximum	Minimum
Copper, per cent	60	55
Zinc, per cent	42	38
Tin, per cent	1.5	0.0
Manganese, per cent	3.5	0.0
Aluminum, per cent	1.5	0.0
Iron, per cent	2.	0.0
Lead, per cent	0.4	0.0

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 143.

[3] 1924 Book of A.S.T.M. Standards.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests. 4. The bronze should conform to the following minimum requirements as to tensile properties:

Tensile strength	65,000 lb. per sq. in.
Elongation in 2 in.	25 per cent

Tension Test Specimen. 5. (*a*) The tension test specimen shall be machined from coupons to the dimensions as shown in Fig. 1.

(*b*) The coupon attached to the castings shall be in accordance with Fig. 2, unless special authority to use coupons of other forms and dimensions is obtained from the purchaser.

(*c*) Each casting weighing 250 lb. or more as delivered from the foundry shall have at least two test coupons cast attached.

(*d*) When the castings constituting a heat weigh less than 250 lb. each, one coupon shall be cast attached to the first and one to the last casting poured in each heat.

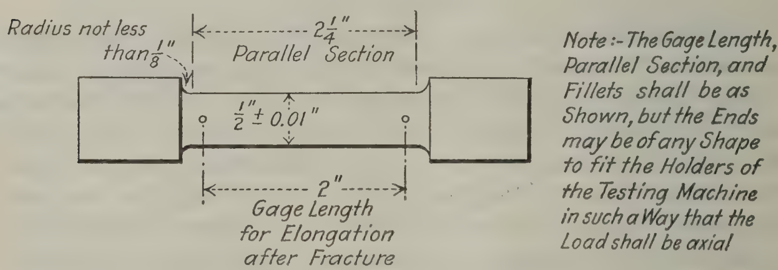

FIG. 1.—Test Specimen.

(*e*) Castings which are too small to permit the casting of a coupon attached shall, when possible, be represented by coupons cast in the same flask and gated to the castings or runners. One coupon shall be cast gated to the first and one to the last casting poured in the heat.

(*f*) Only in cases when it is impossible to cast coupons in the same flask with small castings shall such coupons be cast separately. In case it is necessary to cast coupons separately, one coupon shall be cast to the first of the heats and one to the last.

(*g*) At least two ounces of drillings from each coupon tested shall be forwarded for chemical analysis.

IV. MARKING

Marking. 6. Each casting shall be clearly stamped with the heat number, and the heat number shall also be stamped on the test coupon representing that heat.

V. INSPECTION AND REJECTION

7. (*a*) Inspection may be made at the manufacturer's works where the castings are made or at the point at which they are received at the option of the purchaser. **Inspection.**

(*b*) If the purchaser elects to have inspection made at the manufacturer's works, the inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with

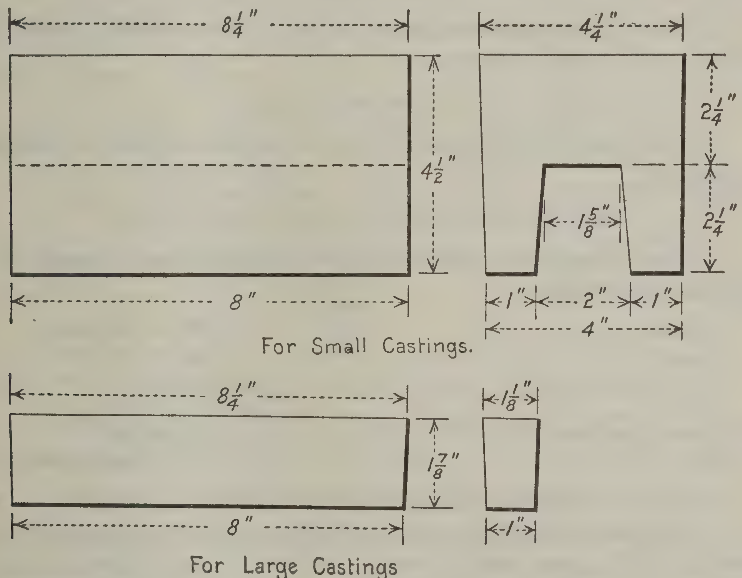

FIG. 2

these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

8. (*a*) Any casting may be rejected upon the failure of the test coupon to meet the physical requirements specified or for the non-compliance with the chemical composition. If the physical or chemical tests indicate that the heat of castings represented by its test coupon does not conform to the physical or chemical requirements, or both, the entire heat shall be rejected. **Rejection.**

(*b*) In case tests on the first test piece do not conform to these specifications, a second test piece shall be tested. If such a test on the second piece is thoroughly satisfactory the castings or heat shall be accepted; if unsatisfactory, the castings or heat shall be rejected.

TENTATIVE SPECIFICATIONS

FOR

MUNTZ METAL CONDENSER TUBE PLATES[1]

Serial Designation: B 57 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Material Covered. 1. These specifications cover rolled Muntz metal plates for surface condenser tube sheets.

I. MANUFACTURE

Process. 2. The plates shall be made by hot rolling from castings of suitable dimensions.

II. CHEMICAL PROPERTIES AND TESTS

Chemical Composition. 3. The plates shall conform to the following requirements as to chemical composition:

Copper	58.00 – 61.00 per cent
Lead	0.35 – 0.90 "
Iron	not over 0.15 "
Tin	not over 0.25 "
Other Impurities	not over 0.10 "
Zinc	Remainder

Chemical Analysis. 4. Analysis may be made on each lot of 5000 lb. or less.

Sampling. 5. The sample shall be taken by drilling or milling representative plates in such a way that a sample of the entire thickness shall be obtained. Equal quantities from each plate shall be thoroughly mixed. Samples so prepared shall be divided into three equal parts, one for the seller, one for the purchaser, and one for an umpire, if necessary.

III. PHYSICAL PROPERTIES AND TESTS

Tension Tests. 6. The material shall conform to the following minimum requirements:

Tensile Strength, lb. per sq. in	Yield Point, lb. per sq. in	Elongation in 2 in., per cent
50 000	20 000	35.0

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

7. The bend test specimen shall stand being bent cold through 120 deg. around a pin, the diameter of which is equal to twice the thickness of the specimen, without cracking on the outside of the bent portion. **Bend Test.**

8. Tension test specimens may be 1 in. in width and of the full thickness of the plate as rolled, or may be machined to conform to the dimensions shown in Fig. 1, in which case the ends shall be of a form to fit the holders of the testing machine in such a way that the load shall be axial. **Tension Test Specimens.**

9. Bend test specimens shall be 0.5 by 1 in. in section with corners rounded to a radius not over $\frac{1}{16}$ in. In the case of thin plates, the specimen may have the same thickness as the plate, with corners rounded to a radius equal to one-fourth of the thickness of the plate. **Bend Test Specimens.**

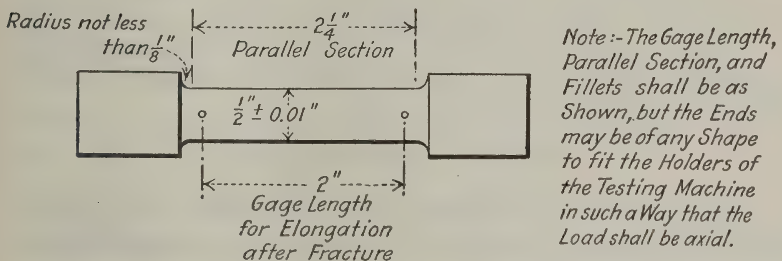

FIG. 1

IV. PERMISSIBLE VARIATIONS IN DIMENSIONS

10. The thickness shall not vary from that specified by more than plus 3 per cent or minus 2 per cent. **Permissible Variations.**

V. WORKMANSHIP AND FINISH

11. The plates shall be free from injurious defects and shall have a smooth clean surface. The fracture shall be uniform in color and grain throughout. **Finish.**

VI. INSPECTION AND REJECTION

12. The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. **Inspection.**

13. Material which fails to conform to these specifications will be rejected and the manufacturer shall be notified. **Rejection.**

TENTATIVE SPECIFICATIONS

FOR

NON-FERROUS ALLOYS FOR RAILWAY EQUIPMENT IN INGOTS, CASTINGS, AND FINISHED CAR AND TENDER BEARINGS.[1]

Serial Designation: B 17 – 21 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1917; REVISED, 1918, 1921.

Material Covered. 1. (*a*) These specifications cover the various non-ferrous alloys for locomotive equipment in ingots, castings and finished car and tender bearings.

(*b*) These alloys and the purposes for which they are used are as follows:

Phosphor Bronze, for connecting-rod bearings, bushings, eccentric straps, crosshead gibs, and miscellaneous bushings, in place of Bearing Metal No. 1, when specifically covered in the contract;

Bearing Metal No. 1, for connecting-rod bearings, bushings, eccentric straps, crosshead gibs and miscellaneous bushings;

Bearing Metal No. 2, for driving-box bearings, engine-truck and trailer bearings, and hub liners;

Bearing Metal No. 3, for lead-lined bearings, for locomotive tenders, freight and passenger-car equipment;

Bell Metal, for locomotive bells only;

Babbitt Metal, for babbitting driving boxes, rod brasses, crossheads, and for hub liners;

Lining Metal, for lining truck brasses, tender brasses and car brasses.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

I. CHEMICAL PROPERTIES AND TESTS.

Chemical Composition.

2. (*a*) The alloys shall conform to the following requirements as to chemical composition, with the permissible variations specified:

Alloy.	Copper, per cent.	Tin, per cent.	Lead, per cent.	Zinc, max., per cent.	Iron, max., per cent.	Antimony, per cent.	Phosphorus, max., per cent.	Sulfur max., per cent.	Arsenic, max., per cent.	Total Impurities, including Zinc, max., per cent.
Phosphor Bronze......	remainder	9–11	8–11	0.75	0.25	0.25*a*	0.7–1.0	....	....	1.0
Bearing Metal No. 1........	remainder	9–11	9–11	1.50	0.25	0.25*a*	....	....	....	2.0
Bearing Metal No. 2........	remainder	4–6	22.5–26.5	0.75	0.40	0.50*a*	....	....	....	1.5
Bearing Metal No. 3........	remainder	4–6	17–22	2.50	0.40	0.50*a*	....	....	....	3.0
Bell Metal.....	remainder	16–18	0.25*a*	0.25	0.25	0.25*a*	0.02*a*	0.05	....	0.50
Babbitt Metal.	0.50*a*	9.25-10.75	remainder	none	....	14–16	....	....	0.20	0.75*b*
Lining Metal...	0.50*a*	4.50-5.50	remainder	none	....	9.25-10.75	....	....	0.20	0.75*b*

a Maximum.
b Must not contain zinc.

(*b*) Where no figures are given for impurity maximum, these elements may be present up to any amount to come within the total impurity specified, provided the material otherwise meets the chemical and physical tests. Where maximum percentages are specified, the elements referred to are considered as impurities and shall not be intentionally added.

Chemical Analysis.

3. An analysis will be made by the purchaser or his representative from one ingot, casting or journal bearing representing each lot. The chemical composition thus determined shall conform to the requirements specified in Section 2 for the particular alloy.

Sample for Chemical Analysis.

4. (*a*) *Ingots.*—Drillings shall be taken with a drill at least ½ in. in diameter at a point midway between the heaviest and smallest cross-section of the ingot. For ingots having two or more sections, drillings shall be taken from each section. In the case of Babbitt Metal and Lining Metal, drillings so taken shall be melted in a thoroughly cleaned ladle with a little rosin flux and poured into a piece of thin section. This piece shall be sawed into three parts and the sawings therefrom thoroughly mixed to constitute the sample for chemical analysis.

(*b*) *Locomotive Castings.*—Drillings shall be taken with a drill as large as possible at such points in the casting as shall be thoroughly representative. Drillings through the skin shall be rejected. Drillings thoroughly mixed shall constitute the sample for chemical analysis.

(*c*) *Car and Tender Bearings.*—Drillings shall be taken with a drill at least ½ in. in diameter, at three points as widely separated as possible on the bearing selected for physical test as specified in Section 5 (*b*). Drillings representing the skin of the casting shall be rejected. These drillings, thoroughly mixed, shall constitute the sample for chemical analysis.

Drillings shall be taken in a like manner from the lining, exercising care that the drill does not penetrate the soldered surface.

(*d*) All samples, whether taken with a drill or saw, shall be kept free of oil and shall be carefully treated with a magnet to remove any iron introduced in taking the sample.

II. PHYSICAL INSPECTION AND TESTS.

Physical Inspection. 5. (*a*) *Locomotive Castings.*—The castings shall be sound, free from blowholes, flaws or shrinkage cracks, and shall show good foundry practice and workmanship.

One casting representing each lot of locomotive bearings will be broken by the purchaser or his representative for examination of fracture. The fracture shall disclose no shrink discoloration, segregation, dross or dirt spots within ⅝ in. of the bored journal engaging surface, and shall show no distinct signs of imperfect mixture.

(*b*) *Car and Tender Bearings.*—The bearings shall be sound, free from sand, blowholes, flaws or shrinkage cracks, and shall show good foundry practice and workmanship.

One bearing representing each lot will be broken by the purchaser or his representative for examination of fracture. The fracture shall show no shrink discoloration, segregation, dross or dirt spots within ½ in. of the bored journal engaging surface. This test shall be made before linings are applied. A second test shall be made after application of the lining as follows:

(1) The suspended brass when tapped with a hammer shall give a distinct ring.

(2) The lining when chipped with a cold chisel shall tear from the soldered surface as distinguished from clean shearing.

III. LOT.

6. (*a*) *Ingots.*—Each 5000 lb. or fraction thereof shall constitute a lot. The ingot from which the sample is taken shall be included in the shipment. **Lot.**

(*b*) *Locomotive Castings.*—Unless otherwise specified, each 100 castings or fraction thereof shall constitute a lot.

The manufacturer shall furnish, free of cost, one casting representing each lot for purpose of tests as specified in Sections 4 (*b*) and 5 (*a*).

(*c*) *Car and Tender Bearings.*—Unless otherwise specified, each 300 bearings or fraction thereof from each pattern shall constitute a lot.

The manufacturer shall furnish, free of cost, one bearing representing each lot for purpose of tests as specified in Sections 4 (*c*) and 5 (*b*).

IV. PERMISSIBLE VARIATIONS IN DIMENSIONS.

(FOR CAR AND TENDER BEARINGS ONLY.)

7. (*a*) The purchaser will furnish drawings showing the dimensions of the bearings ordered and permissible variations therefrom, and the bearings shall conform to these drawings within such permissible variations. **Permissible Variations.**

(*b*) The thickness at the center of the brass and of the complete bearing shall not vary more than $\frac{1}{32}$ in. over or under the normal thickness shown on the drawing. The thickness of the lining shall not vary more than $\frac{1}{32}$ in. over or under the normal thickness shown on the drawing.

V. WORKMANSHIP.

8. (*a*) *Locomotive Castings.*—The castings shall conform to the dimensions specified and shall show good workmanship generally. **Workmanship.**

(*b*) *Car and Tender Bearings.*—The bearings shall show good workmanship generally, and shall be free from mechanical imperfections. They shall be bored to a true radius, and with the axis parallel to the plane of the top and perpendicular to the side lugs and bearing flange. The lettering shall be clear and distinct. No emery shall be used on journal engaging surfaces on either the brass or the lining.

VI. MARKING.

Marking. 9. (*a*) *Ingot.*—The manufacturer's marking only shall be required, unless otherwise specified.

(*b*) *Locomotive Castings.*—The lot number and other marks as required by the drawings shall be legibly marked on each casting.

(*c*) *Car and Tender Bearings.*—The name or initials of the manufacturer, the initials of the purchaser, type number, journal size, and lot number shall be legibly cast with raised figures on a depressed surface of each bearing, as shown on the drawings.

VII. INSPECTION AND REJECTION.

Inspection. 10. (*a*) Inspection may be made at the manufacturer's works where the ingots, locomotive castings or car and tender bearings are made, or at the point at which they are received, at the option of the purchaser.

(*b*) If the purchaser elects to have inspection made at the manufacturer's works, the inspector representing the purchaser shall have free entry at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, free of cost, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection. 11. Failure to meet the chemical or physical specifications, or non-conformity to any of the above requirements as to permissible variations, dimensions and markings, constitute sufficient cause for rejection of the lot represented by the sample chosen.

TENTATIVE SPECIFICATIONS

FOR

WHITE METAL BEARING ALLOYS.

(KNOWN COMMERCIALLY AS "BABBITT METAL.")[1]

Serial Designation: B 23 – 18 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1918.

Material Covered. 1. These specifications cover white metal bearing alloys, known commercially as "babbitt metal." Twelve typical babbitt metals are specified, covering the range of alloys commercially used and are designated as Grades Nos. 1 to 12, in accordance with their decreasing tin content as specified in Section 4.

I. MANUFACTURE.

Uniform Quality. 2. The manufacturer shall use care to have each lot of babbitt metal as uniform in quality as possible.

Dimensions. 3. The standard bar, unless otherwise specified, shall have the following approximate dimensions:

Top face	$1\frac{1}{4}$ in. wide, $8\frac{1}{2}$ in. long.
Thickness	not over $1\frac{1}{4}$ in.

II. CHEMICAL PROPERTIES AND TESTS.

Chemical Composition. 4. The alloys shall conform to the following requirements as to chemical composition, within the limits specified in Section 5:

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

Alloy, Grade No.	Tin, per cent.	Antimony, per cent.	Lead, per cent.	Copper, per cent.	Iron, max., per cent.	Arsenic, max., per cent.	Zinc, per cent.	Aluminum, per cent.
1........	91	$4\frac{1}{2}$	0.35[a]	$4\frac{1}{2}$	0.08	0.10	none	none
2........	89	$7\frac{1}{2}$	0.35[a]	$3\frac{1}{2}$	0.08	0.10	none	none
3........	$83\frac{1}{3}$	$8\frac{1}{3}$	0.35[a]	$8\frac{1}{3}$	0.08	0.10	none	none
4........	75	12	10	3	0.08	0.15	none	none
5........	65	15	18	2	0.08	0.15	none	none
6........	20	15	$63\frac{1}{2}$	$1\frac{1}{2}$	0.08	0.15	none	none
7........	10	15	75	0.50[a]	....	0.20	none	none
8........	5	15	80	0.50[a]	....	0.20	none	none
9........	5	10	85	0.50[a]	....	0.20	none	none
10........	2	15	83	0.50[a]	....	0.20	none	none
11........	..	15	85	0.50[a]	....	0.25	none	none
12........	..	10	90	0.50[a]	....	0.25	none	none

[a] Maximum.

Permissible Variations.

5. The following permissible variations in the percentages of the desired elements specified in Section 4 will be allowed, but shall not apply to the maximum percentages of impurities specified:

PERCENTAGE OF ELEMENT SPECIFIED.	PERMISSIBLE VARIATIONS OVER OR UNDER THE SPECIFIED VALUE, UNITS OF PER CENT.
Not over 2 per cent	0.25
Over 2 to 5 per cent, incl	0.50
Over 5 to 10 per cent, incl	0.75
Over 10 per cent	1.00

Samples for Chemical Analysis.

6. (*a*) Three bars shall be selected to represent a shipment of less than 1000 lb., five bars to represent a shipment of over 1000 lb. to 10,000 lb., inclusive, and ten bars to represent a shipment of over 10,000 lb. to one carload.

(*b*) Saw cuts shall be made at points in the bars indicated in Fig. 1. No lubricants shall be used for sawing. The sawings shall be carefully treated with a magnet to remove any particles of steel introduced in taking the sample.

(*c*) Sawings thoroughly mixed shall constitute the sample for chemical analysis.

Methods of Chemical Analyses.

7. The chemical analysis shall be made in accordance with the Standard Methods of Chemical Analysis of Alloys of Lead, Tin, Antimony and Copper (Serial Designation: B 18) of the American Society for Testing Materials.[1]

[1] 1924 Book of A.S.T.M. Standards.

III. MARKING.

8. The name of the manufacturer shall be cast on each bar. The numerical designation of the grade supplied shall be stamped or cast on each bar for identification. **Marking.**

NOTE.—A preferred arrangement of marking is shown in Fig. 2.

IV. CLAIMS.

9. Claims, to be considered, shall be made in writing within thirty days of receipt of material at the purchaser's plant, and **Claims.**

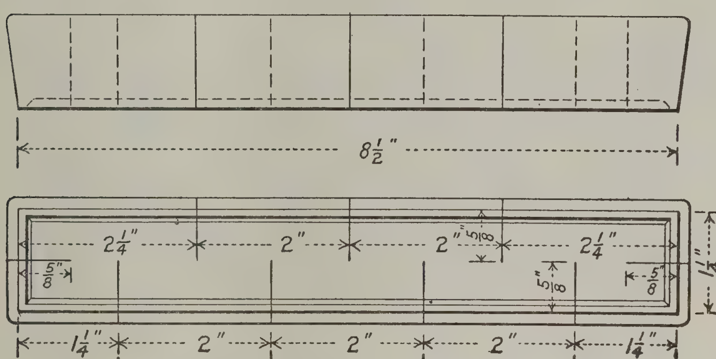

FIG. 1.—Method of Sampling.

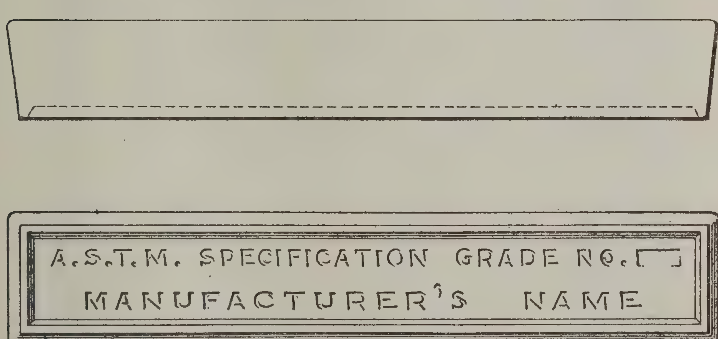

FIG. 2.—Preferred Arrangement of Marking.

the results of the purchaser's tests shall be given. The shipper shall within one week of receipt of such claim, either agree to satisfy the claim or send a representative to the purchaser's plant to resample the shipment, as specified in Section 6. Samples so taken shall be sealed and submitted to a mutually agreeable umpire, whose determination shall be final.

Settlement of Claims.

10. The expense of umpire analysis shall be paid by the loser, or divided in proportion to the concession made in case of a compromise. In case of rejection being established, the damages shall be limited to the payment of freight both ways by the manufacturer for the substitution of an equivalent weight of babbitt metal meeting these specifications.

APPENDIX.

The data in the following table do not constitute a part of these specifications. They are given merely to indicate to the purchaser the physical properties of the various alloys specified which can be expected of carefully manufactured alloys of the formulas indicated, and to constitute a guide to the purchaser in selecting the grade best suited for meeting the service condition for which the babbitt metal is to be used. The alloys were prepared at the U. S. Bureau of Standards from pure Banka tin, high grade "Star" antimony and a commercially pure lead (99.94 per cent Pb), and the values given are the results of tests made in the Bureau laboratories. These figures are subject to revision.

COMPOSITION AND PHYSICAL PROPERTIES[1] OF WHITE METAL BEARING ALLOYS

Alloy Number	Specified Composition of Alloys				Specific Gravity	Compositions of Alloys Tested				Yield Point, lb. per sq. in.[2]		Johnson's Apparent Elastic Limit, lb. per sq. in.[3]		Ultimate Strength, lb. per sq. in.[4]		Brinell Hardness[5]		Melting Point		Temperature of Complete Liquidation		Proper Pouring Temperature	
	Copper, per cent	Tin, per cent	Antimony, per cent	Lead, per cent		Copper, per cent	Tin, per cent	Antimony, per cent	Lead, per cent	20° C.	100°C.	20° C.	100°C.	20° C.	100°C.	20° C.	100°C.	Deg. Fahr.	Deg. Cent.	Deg. Fahr.	Deg. Cent.	Deg. Fahr.	Deg. Cent.
1	4.5	91.0	4.5	....	7.34	4.56	90.9	4.52	none	4400	2650	2450	1050	12850	6950	17.0	8.0	433	223	...	...	825	441
2	3.5	89.0	7.5	....	7.39	3.1	89.2	7.4	0.03	6100	3000	3350	1100	14900	8700	24.5	12.0	466	241	669	354	795	424
3	8⅓	83⅓	8⅓	....	7.46	8.3	83.4	8.2	0.03	6600	3150	5350	1300	17600	9900	27.0	14.5	464	240	792	422	915	491
4	3.0	75.0	12.0	10.0	7.52	3.0	75.0	11.6	10.2	5550	2150	3200	1550	16150	6900	24.5	12.0	363	184	583	306	710	377
5	2.0	65.0	15.0	18.0	7.75	2.0	65.5	14.1	18.2	5050	2150	3750	1500	15050	6750	22.5	10.0	358	181	565	296	690	366
6	1.5	20.0	15.0	63.5	9.33	1.5	19.8	14.6	63.7	3800	2050	3550	1800	14550	8050	21.0	10.5	358	181	531	277	655	346
7	...	10.0	15.0	75.0	9.73	0.11	10.0	14.5	75.0	3550	1600	2500	1350	15650	6150	22.5	10.5	464	240	514	268	640	338
8	...	5.0	15.0	80.0	10.04	0.14	5.2	14.9	79.4	3400	1750	2650	1200	15600	6150	20.0	9.5	459	237	522	272	645	341
9	...	5.0	10.0	85.0	10.24	0.06	5.0	9.9	84.6	3400	1550	2400	950	14700	5850	19.0	8.5	459	237	493	256	620	327
10	...	2.0	15.0	83.0	10.07	0.12	2.05	15.7	82.0	3350	1850	2250	1200	15450	5750	17.5	9.0	468	242	507	264	630	332
11	...	....	15.0	85.0	10.28	0.19	0.09	14.8	84.7	3050	1400	2750	1100	12800	5100	15.0	7.0	471	244	504	262	630	332
12	...	....	10.0	90.0	10.67	0.12	0.11	9.9	89.4	2800	1250	2250	950	12900	5100	14.5	6.5	473	245	498	259	625	329

[1] The compression test specimens were cylinders $1\frac{1}{2}$ in. in length and $\frac{1}{2}$ in. diameter, machined from chill castings 2 in. in length and $\frac{3}{4}$ in. in diameter. The Brinell tests were made on the bottom face of parallel machined specimens cast in a 2-in. diameter by $\frac{5}{8}$-in. deep steel mold at room temperature.

[2] The values for yield point were taken from stress-strain curves at a deformation of 0.125 per cent reduction of gage length.

[3] Johnson's apparent elastic limit is taken as the unit stress at the point where the slope of the tangent to the curve is $1\frac{1}{2}$ times its slope at the origin.

[4] The ultimate strength values were taken as the unit load necessary to produce a deformation of 25 per cent of the length of the specimen.

[5] These values are the average Brinell number of three impressions on each alloy using a 10-mm. ball and a 500-kg. load applied for 30 seconds.

TENTATIVE SPECIFICATIONS
FOR
ALUMINUM INGOTS FOR REMELTING[1]

Serial Designation: B 24 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1918; REVISED, 1919, 1920, 1922, 1925.

Material Covered.

1. These specifications cover three grades of Virgin Aluminum, as follows:

GRADE.	ALUMINUM, PER CENT.
99.5	99.5 (Minimum)
99.0	99.0 (Minimum)
98.0	98.0 (Minimum)

Impurities.

2. The presence in any of the above grades of manganese, magnesium, zinc, calcium or similar metals commonly used in the light alloys, is not admissible and such ingot cannot be classed as Virgin Aluminum under these specifications. Copper is an allowable impurity up to a maximum of 0.1 per cent in the 99.5 grade; 0.25 per cent in the 99.0 grade; and 0.45 per cent in the 98.0 grade. Iron and silicon are also allowable impurities. The sum total of the impurities, however, shall not exceed 0.5 per cent in the 99.5 grade, 1.0 per cent in the 99.0 grade, nor 2.0 per cent in the 98.0 grade.

I. MANUFACTURE.

Manufacture.

3. No scrap shall be used other than that resulting in the manufacturer's own plant from the production of material of composition similar to that specified.

II. PHYSICAL DEFECTS.

Physical Defects.

4. Aluminum ingots shall have a clean surface and shall be free from dirt, slag and foreign material.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

III. SAMPLING.

5. If the aluminum is shipped in carload lots, then random samples shall be taken, but not less than five ingots for each car. If the shipment is in less than carload lots, one ingot shall be taken for each 10,000 lb. (4536 kg.) or fractional part thereof. **Sampling.**

When it is deemed necessary, a sample may be taken from each heat of 500 lb. (226.8 kg.) or more of metal.

6. The ingots selected shall be drilled without lubricant completely through, or half way through from top and bottom. The drillings shall be fine, thin chips. They should be thoroughly mixed and the sample for analysis should not be less than 60 g. **Drilling.**

IV. ANALYSES.

7. The chemical analysis shall be made in accordance with the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (Serial Designation: B 40 – 25 T) of the American Society for Testing Materials.[1] **Analyses.**

V. MARKING.

8. The maker's brand and the grade of aluminum shall be cast on each ingot. When this is not practicable, the grade shall be indicated by spotting each large ingot, or one ingot in each bundle of small ingots, with a designating color of paint: white indicating the 99.5 grade, blue, the 99.0 grade, and red, the 98.0 grade. **Marking.**

VI. CLAIMS.

9. Claims, to be considered, shall be made in writing within thirty days of receipt of material at the purchaser's plant, and the results of the purchaser's tests shall be given. The shipper shall within one week of receipt of such claim, either agree to satisfy the claim or send a representative to the purchaser's plant to resample the shipment, as specified in Section 3. Samples so taken shall be sealed and submitted to a mutually agreeable umpire, whose determination shall be final. **Claims.**

10. The expense of the umpire analysis shall be paid by the loser, or divided in proportion to the concession made in case of a compromise. In case of rejection being established, the damage shall be limited to the payment of freight both ways by the manufacturer for the substitution of an equivalent weight of metal meeting these specifications. **Settlement of Claims.**

[1] See p. 171.

TENTATIVE SPECIFICATIONS

FOR

ALUMINUM SHEET.[1]

Serial Designation: B 25 – 19 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1918; REVISED, 1919.

I. MANUFACTURE.

Manufacture. 1. No scrap shall be used in the manufacture of aluminum sheet except such as shall accumulate at the manufacturer's own plant from material of the same composition and of his own manufacture.

II. CHEMICAL PROPERTIES AND TESTS.

Chemical Analyses. 2. The sheet may be rolled from aluminum ingots of 99.0 grade, described in the Tentative Specifications for Aluminum Ingots for Remelting (Serial Designation: B 24 – 25 T) of the American Society for Testing Materials.[2]

3. Samples for analysis shall be obtained from a random sheet, representing each 500 lb. (226.8 kg.) of aluminum or any lot weighing less than 500 lb. (226.8 kg.), as agreed upon between the seller and the purchaser. Samples so prepared shall be divided into three equal parts, each of which shall be placed in a sealed package, one for each party and one for an umpire if necessary. The sample for analysis may be prepared by shearing.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 160.

III. PHYSICAL PROPERTIES AND TESTS.

4. (*a*) Sheets may be furnished in either of three tempers or degrees of hardness and shall conform to the following requirements as to tensile properties: **Tension Tests.**

B. & S. Gage.	Thickness, in.	Minimum Tensile Strength, lb. per sq. in.	Minimum Elongation in 2 in., per cent.
TEMPER No. 1; SOFT, ANNEALED.			
12 to 16, incl.	0.0808 - 0.0509	12 500	30
17 " 22, "	0.0508 - 0.0227	12 500	20
23 " 26, "	0.0226 - 0.0159	12 500	10
TEMPER No. 2; HALF-HARD.			
12 to 16, incl.	0.0808 - 0.0509	18 000	7
17 " 22, "	0.0508 - 0.0227	18 000	5
23 " 26, "	0.0226 - 0.0159	18 000	5
TEMPER No. 3; HARD.			
12 to 16, incl.	0.0808 - 0.0509	22 000	4
17 " 22, "	0.0508 - 0.0227	25 000	2
23 " 26, "	0.0226 - 0.0159	30 000	2

(*b*) The tension test specimen shall be taken parallel to the direction of cold rolling of the sheet.

5. Sheets of temper No. 1 shall withstand being bent double in any direction and hammered flat, and sheets of temper No. 2, being bent around a pin of radius equal to the thickness of the sheet, without cracking. **Bend Tests.**

6. (*a*) One tension and one bend test specimen shall be cut from a random sheet representing each 500 lb. (226.8 kg.) of aluminum or any lot weighing less than 500 lb. (226.8 kg.), as agreed upon between the seller and the purchaser. **Number of Tests.**

(*b*) Test specimens may be used for purposes of analysis specified in Section 3.

IV. PERMISSIBLE VARIATIONS IN DIMENSIONS.

7. The thickness of sheets shall not vary from that specified by more than the following amounts: **Dimensional Variations.**

B. & S. Gage.	Thickness, in.	Permissible Variation, in.
10 to 17, incl.	0.1019 - 0.0404	0.003
18 " 26, "	0.0403 - 0.0159	0.002

V. WORKMANSHIP AND FINISH.

Workmanship. 8. All sheets shall be commercially flat and free from buckles; they shall be free from injurious surface defects and shall have a workmanlike finish.

VI. INSPECTION AND REJECTION.

Inspection. 9. The manufacturer shall afford the inspector representing the purchaser, free of cost, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications.

Rejection. 10. Material which fails to conform to these specifications will be rejected and the manufacturer shall be notified.

Explanatory Notes.

Section 4.—Aluminum sheet cut in other directions than parallel to the direction of cold rolling will show a ductility less than that in this direction; the elongation at right angles to the direction of cold rolling may be only two-thirds of the former.

It is strongly recommended that a self-centering tension test specimen holder be used for testing particularly thin gages of hard aluminum sheet, in view of the lack of uniformity which may occur in test series made in the usual wedge grips.

TENTATIVE SPECIFICATIONS
FOR
ALUMINUM FOR USE IN THE MANUFACTURE OF IRON AND STEEL.[1]

Serial Designation: B 37 – 20 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1920.

Material Covered.

1. These specifications cover two grades of Virgin and four grades of Secondary Aluminum, as follows:

(*a*) *Virgin Aluminum.*

(1) *98.0 Grade*, containing not less than 98 per cent aluminum. This grade is identical with the 98.0 Grade specified in the Tentative Specifications for Aluminum Ingots for Remelting (Serial Designation: B 24 – 25 T) of the American Society for Testing Materials.[2]

(2) *94.0 Grade*, containing not less than 94 per cent aluminum. The remainder may be chiefly iron and silicon.

(*b*) *Secondary Aluminum.*

(1) *98.0 Grade*, containing not less than 98 per cent aluminum. This grade is generally made from sheet clippings. The remainder may be chiefly copper, iron and silicon.

(2) *94.0 Grade*, containing not less than 94 per cent aluminum. A maximum of 0.5 per cent of zinc will be allowed in this grade. The remainder may be chiefly copper.

(3) *90.0-A Grade*, containing not less than 90 per cent aluminum. A maximum of 0.5 per cent of zinc will be allowed in this grade. The remainder may be chiefly copper.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 160.

(4) *90.0-B Grade*, containing not less than 90 per cent aluminum. A maximum of 4 per cent of zinc will be allowed in this grade. The remainder may be chiefly copper.

I. MANUFACTURE.

Manufacture. 2. The manufacturer shall use care to have each lot of metal as uniform as possible.

3. The aluminum may be in the form of ingots, rods or shot.

Ingots should be of convenient size for use. Ingots with a minimum of five notches, weighing approximately 3 lb. will meet the requirements of these specifications.

Rods may be either rolled or cast and $\frac{3}{8}$ to $\frac{1}{2}$ in. in diameter.

Shot should be of convenient size. That produced by running the molten metal through $\frac{1}{8}$-in. diameter holes is satisfactory.

II. SAMPLING.

Sampling. 4. If the aluminum is shipped in carload lots, random samples shall be taken but not less than five ingots for each car. If the shipment is in less than carload lots, one ingot shall be taken for each 10,000 lb. (4536 kg.) or fractional part thereof.

Drilling. 5. The ingots selected shall be drilled completely through, or half through from top and bottom. The drillings shall be fine, thin chips. They shall be thoroughly mixed, and the sample for analysis shall not be less than 60 g.

Rods shall be sampled in a similar manner, except that the drilling shall be radial.

An equivalent amount of shot shall be taken as a sample and reduced by quartering to a sample of convenient size.

III. ANALYSES.

Analyses. 6. The chemical analysis shall be made in accordance with the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (Serial Designation: B 40 – 25 T) of the American Society for Testing Materials.[1]

IV. MARKING.

Marking. 7. The maker's brand and the grade of aluminum shall be cast or rolled on each ingot or rod and marked plainly on each container of shot metal. When marking the grade by casting or rolling is not practicable, the grade shall be indicated by

[1] See p. 171.

spotting each large ingot or one ingot or rod from each bundle with a designating color of paint, red indicating the 98.0 Virgin grade, yellow the 94.0 Virgin grade, red and white the Secondary 98.0 Grade, red and yellow the Secondary 94.0 Grade, red and blue the Secondary 90.0-A Grade, and red and black the Secondary 90.0-B Grade.

V. CLAIMS.

8. Claims, to be considered, shall be made in writing within ten days of receipt of material at the purchaser's plant, and the results of the purchaser's tests shall be given. The vender shall within ten days of such claim, either agree to satisfy the claim or send a representative to the purchaser's plant to resample the shipment as specified in Section 3. Samples so taken shall be sealed and submitted to a mutually agreeable umpire, whose determination shall be final. **Claims.**

10. The expense of the umpire analysis shall be paid by the loser, or divided in proportion to the concession made, in case of compromise. In case rejection is established, the damage shall be limited to the payment of freight both ways by the vender for the substitution of an equivalent weight of metal meeting specifications.

TENTATIVE SPECIFICATIONS
FOR
NON-FERROUS INSECT SCREEN CLOTH[1]

Serial Designation: B 50 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1925.

Scope. 1. These specifications cover insect screen cloth woven from non-ferrous wire and intended to be used as a protection against insects or other small animals.

I. MANUFACTURE

Manufacture. 2. There shall be two classes of standard insect screen cloth depending upon the material used as follows:

(*a*) *Class A*, comprising cloth made from copper wire.

(*b*) *Class B*, comprising cloth made from a 90 copper-10 zinc alloy wire.

3. Insect screen cloth shall be manufactured in four weights or sizes of mesh as follows:

14-mesh	16-mesh heavy
16-mesh	18-mesh

4. The same quality and size of wire shall be used for both the warp and filling.

II. CHEMICAL PROPERTIES

Chemical Requirements. 5. The composition of the wire used for Class A insect screen cloth shall conform to the requirements either of the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 4), or of the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (Serial Designation: B 5) of the American Society for Testing Materials.[2]

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. W. H. Bassett, Secretary of Committee D-14 on Screen Wire Cloth, American Brass Co., Waterbury, Conn.

[2] 1924 Book of A.S.T.M. Standards.

6. The wire for Class B insect screen cloth shall conform to the following chemical requirements:

Copper	90.00 to 92.00 per cent
Lead, maximum	0.03 per cent
Iron, maximum	0.06 per cent
Zinc	Remainder

III. PHYSICAL PROPERTIES OF WIRE

7. The physical properties for all classes of screen wire shall be subject to agreement between the producer of the wire and the purchaser.

NOTE.—Wire for weaving insect screen cloth is usually furnished with a drawn temper. The needs of different weavers of wire cloth apparently call for difference in drawn temper and it is consequently at present impossible to give definite physical properties which would be satisfactory to the entire trade. It has, therefore, seemed better to leave this matter to specific agreement between the producers of wire and the manufacturers of wire cloth.

IV. STANDARD SIZES, WEIGHTS, DIMENSIONS, ETC.

8. (*a*) The nominal diameter of wires to be used for the various weights of standard insect screen cloth shall be in accordance with the following: **Size of Wire.**

Mesh	Diameter, in.
14	0.0119
16	0.0113
16 (Heavy)	0.0150
18	0.0106

(*b*) The permissible variation from the nominal diameter of any of the wires specified above shall be ± 0.00025 in.

9. The mesh as determined by counting the openings per linear inch for a length of one foot on any samples selected at random shall not vary from the nominal value in the warp, but an average variation of not more than $\frac{1}{2}$ mesh per linear inch shall be permissible in the filling.

10. Insect screen cloth shall be furnished in rolls of approximately 100 linear feet. At least 87.5 per cent of the rolls ordered shall be in one continuous length; the remaining rolls may be in two lengths.

11. The widths of standard insect screen cloth shall be from 18 to 48 in. in multiples of 2 in. The permissible variation in over all width as measured from outside wire to outside wire shall be $\pm\frac{1}{8}$ in.

V. WORKMANSHIP AND FINISH

12. (*a*) The insect screen cloth shall be free from all imperfections and blemishes not consistent with best commercial practice.

(*b*) Well-made splices in individual wires are permitted at any point in the screen cloth.

VI. INSPECTION

13. (*a*) All testing and inspection, both of individual wires and completed cloth, shall be made at the place of manufacture. Tests on individual wires shall be made on samples taken before weaving and not on wires removed from finished cloth.

(*b*) The manufacturer shall afford the inspector representing the purchaser all reasonable facilities to satisfy him that the material conforms to these specifications.

Appendix

The following table giving the weights of various mesh of insect screen cloth is not a part of these specifications, but is appended as a source of information for the purchaser:

Mesh	Approximate Weight per 100 sq. ft. of Insect Screen Cloth Wrapped and Labeled Ready for Shipment
14	$14\frac{1}{2}$ lb.
16	15 "
16 (Heavy)	$27\frac{3}{4}$ "
18	14 "

TENTATIVE METHODS OF CHEMICAL ANALYSIS
OF
ALUMINUM AND LIGHT ALUMINUM ALLOYS.[1]

Serial Designation: B 40 – 25 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1923, 1925.

The methods of analysis listed under "Analysis of Aluminum" contemplate the analysis of aluminum containing more than 98 per cent aluminum and containing only silicon, titanium, iron, copper, carbon and traces of manganese as impurities.

Unless it is positively known that such elements as tin, antimony, zinc, lead, nickel, magnesium, sodium, nitrogen or oxygen (as aluminum oxide) are absent, the methods listed under the "Analysis of Light Aluminum Alloys" should be employed.

ANALYSIS OF ALUMINUM.

(NOT LESS THAN 98 PER CENT ALUMINUM.)

DETERMINATION OF SILICON AND TITANIUM.

DETERMINATION OF TOTAL SILICON.

SOLUTIONS REQUIRED.

"Acid Mixture."—1200 cc. of H_2SO_4 (25-per-cent by volume), 600 cc. HCl (sp. gr. 1.20) and 200 cc. HNO_3 (sp. gr. 1.42).

25-per-cent Sulfuric Acid.—Pour 250 cc. of H_2SO_4 (sp. gr. 1.84) into distilled water, and dilute to 1000 cc.

METHOD.

Dissolve 1 g. of the well-mixed sample in 35 cc. of the acid mixture in a covered flat-bottomed porcelain dish or casserole. When the drillings are completely dissolved, boil the solution to complete

[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

expulsion of HNO_3 and HCl and heat until copious fumes of H_2SO_4 have been given off for five minutes. Cool and take up the residue with 10 cc. of 25-per-cent H_2SO_4 and 100 cc. of water. Warm to complete solution of the sulfates and filter on an ashless filter paper. Wash the residue with hot water until free from sulfates and ignite in a platinum crucible. Fuse the residue with 8 to 10 times its weight of Na_2CO_3 and treat the fused mass in a casserole or beaker with 20 cc. of H_2SO_4 (1:3), washing out the crucible. Evaporate the solution until it fumes strongly to separate the SiO_2. Cool, dilute to about 100 cc., boil till the salts are dissolved, filter, wash well, ignite in a platinum crucible and weigh. Treat the ash with HF and a few drops of H_2SO_4 (1:3), carefully evaporate till dry, ignite and again weigh.

From the difference between the two weights deduct a blank obtained from the reagents and filter. The rest of the loss in weight represents SiO_2 corresponding to the Si in the metal.

$$SiO_2 \times 0.4672 = Si.$$

Notes.

Blanks should be carried along with all reagents and the proper corrections made.

In routine analysis, fusion of the first H_2SO_4 residue with sodium carbonate and subsequent treatment to the point of treating the ash with HF may be omitted. The first residue may be weighed and treated with HF, etc., as directed for the second H_2SO_4 residue. This, however, should not be done if the material contains much graphitic silicon or if the residue after ignition is dark colored.

DETERMINATION OF TITANIUM.

Apparatus and Solutions Required.

Nessler tubes or some form of colorimeter.

Hydrogen Peroxide U. S. P.—This reagent must be free from HF.

Standard Titanium Solution.—This solution is best prepared and standardized as follows:[1]

"Potassium titanium fluoride, K_2TiF_6, best serves as the starting point for the preparation of the standard solution. A quantity of this is recrystallized from boiling water one or more times, dried, and preserved in a glass-stoppered bottle. Enough of it to make 0.5 to 1 liter of the standard sulfate solution is put into a platinum dish and evaporated several times with strong sulfuric acid without bringing to dryness, till the fluorine is completely expelled. The residue is then taken up with water containing enough sulfuric acid to make at least

[1] W. F. Hillebrand, "The Analysis of Silicate and Carbonate Rocks," *Bulletin No. 700*, U. S. Geological Survey, p. 157.

5 per cent of the latter when fully diluted. Two 50 to 100-cc. portions of the prepared solution are then further diluted, boiled, and precipitated with ammonia. The precipitates are collected on paper, washed with hot water till free from alkali, ignited moist in the filter, blasted, and weighed. Duplicates should agree almost exactly. From the weights found, the strength of the standard is calculated and the result affixed to the bottle containing it. The stopper of the bottle should be coated with vaseline and the needed quantities of solution should be withdrawn by a pipette, never poured. In a solution so prepared there is not enough alkali sulfate to weaken the color when peroxidized."

METHOD.

See "Determination of Total Silicon."

Fuse the non-volatile residue remaining after the silicon determination in a small amount of $K_2S_2O_7$, take up in a little 5-per-cent H_2SO_4 and add to the filtrates and washings from the silicon determination. Evaporate the solution to approximately 100 cc., and treat with 5 cc. H_2SO_4 (sp. gr. 1.84) and 3 g. of iron-free zinc. Heat until the zinc is nearly dissolved and the reduction of copper is complete. Decant the solution into another beaker, wash the zinc and copper with hot water and continue the evaporation of the solution to approximately 75 cc. Cool and transfer to a 100-cc. Nessler's comparison tube, add 5 cc. of 3-per-cent H_2O_2 and dilute to the 100-cc. mark. In another tube, place 88 cc. H_2O, 5 cc. of H_2SO_4 (sp. gr. 1.84) and cool. Add 5 cc. of 3-per-cent H_2O_2 and then from a burette add the standard titanium solution until the colors match. The volume of the standard solution required multiplied by 100 times its titre represents the percentage of titanium in the sample.

NOTES.

The filtrate and washings from the Silicon Determination should ordinarily be colorless. If light yellow owing to iron, the standard comparison solution should be brought to the same tint with ferric sulfate solution before adding the hydrogen peroxide.

In case the amount of titanium is high the solution may be diluted to a definite volume and aliquot portions taken for the colorimetric test.

DETERMINATION OF IRON

SOLUTIONS REQUIRED

"*Acid Mixture.*"—1200 cc. of H_2SO_4 (25-per-cent by volume), 600 cc. of HCl (sp. gr. 1.20) and 200 cc. of HNO_3 (sp. gr. 1.42).

Tartaric Acid Solution.—Dissolve 20 g. of tartaric acid crystals in 100 cc. of distilled water.

Dilute Ammonium Sulfide Solution.—Add 15 cc. of NH_4OH (sp. gr. 0.90) to 15 cc. of distilled water and saturate with H_2S; then dilute to 200 cc. with distilled water. Use freshly made.

Dilute Sulfuric Acid Solution for Reductor (5-per-cent).—Mix 50 cc. of H_2SO_4 (sp. gr. 1.84) and 1000 cc. of distilled water.

Standard Potassium Permanganate Solution.—Dissolve 1 g. $KMnO_4$ in 1000 cc. of H_2O, and allow to stand for several days in a glass-stoppered bottle in a dark closet. Filter the solution through prepared asbestos and standardize against 0.1000-g. portions of pure sodium oxalate. Each cubic centimeter is equivalent to approximately 0.00177 g. of iron.

METHOD

Dissolve 1 g. of the well-mixed sample in 35 cc. of the acid mixture and proceed as in "Determination of Silicon," including treatment with HF omitting the weighing of residues. Fuse the last residue obtained with a small amount of $K_2S_2O_7$, take up in a little 5-per-cent H_2SO_4, and add to the filtrates and washings obtained. Saturate this solution with H_2S and filter. To the filtrate add 25 cc. of the tartaric acid solution, and then with constant stirring, add NH_4OH (sp. gr. 0.90), finally drop by drop, until it is in slight excess. Pass in H_2S for a few moments, warm somewhat and allow the solution to stand. Filter and wash the sulfide precipitate thoroughly with a freshly made dilute ammonium sulfide solution, until all tartaric acid is washed out.

Dissolve the sulfide precipitate through the filter paper with warm dilute H_2SO_4 (1:10) washing the paper thoroughly, alternately with hot distilled water and with 5-per-cent H_2SO_4. Boil until all H_2S is expelled and add HNO_3 (sp. gr. 1.42) until the iron is oxidized, which is shown by the solution becoming of a yellowish color. Add 3 to 4 g. of NH_4Cl and then NH_4OH (sp. gr. 0.90) in excess until a permanent precipitate forms. Boil again, allow to settle and filter, washing with dilute ammonia and hot distilled water.

Dissolve the washed precipitate through the filter with warm dilute H_2SO_4 (1:10), wash the paper thoroughly with 5-per-cent H_2SO_4 and hot distilled water, using in all about 100 cc. Pass the solution through the reductor and wash the beaker and reductor with 150 cc. of dilute H_2SO_4 for reductor and then 100 cc. of distilled water. Titrate with standard $KMnO_4$ solution.

NOTES

Results of titration should be checked with a blank determination made on corresponding amounts of solutions used.

A small quantity of liquid should always be left in the reductor funnel and air should never be allowed to enter the body of the reductor.

For further details of the use of reductor see "The Chemical Analysis of Iron," by A. S. Blair, or "Quantitive Chemical Analysis," by H. P. Talbot.

The effect of tartaric acid is to form complexes with aluminum, vanadium, chromium, titanium, etc., preventing their precipitation as hydroxides.

Where the iron exists in very small quantities, a gravimetric method is preferable. In this case, double precipitation of iron sulfide is necessary to eliminate all aluminum. Dissolve the second sulfide precipitate in warm dilute H_2SO_4 (1:10), boil, and oxidize the iron with H_2O_2. Add 3 to 4 g. of NH_4Cl and then NH_4OH (sp. gr. 0.90) in considerable excess, with stirring. Filter quickly, dry and ignite the precipitate, weighing as ferric oxide. If an accurate determination of iron is desired, it is necessary to correct for the small accumulation of silica by treatment of the ignited precipitate with H_2SO_4. HF.

DETERMINATION OF COPPER BY ELECTROLYTIC METHOD

APPARATUS FOR ELECTROLYSIS

See the "Determination of Copper by the Electrolytic Method" in the Standard Methods of Chemical Analysis of Manganese Bronze (Serial Designation : B 27) of the American Society for Testing Materials.[1]

SOLUTIONS REQUIRED

Sodium Hydroxide Solution.—Dissolve 250 g. of NaOH in distilled water and dilute to 1000 cc.

Nitric Acid (1:1)—Pour 500 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 1000 cc.

METHOD

Dissolve 10 g. of the sample in a large casserole or beaker with 150 cc. of the NaOH solution. This should be added in small portions as the action is vigorous for a time. After the action slackens, complete solution may be hastened by warming. When no evidence of further reaction can be seen, dilute to about 700 cc. with water at the boiling temperature. Filter through a strong paper and wash.

Dissolve the residue from the paper with 25 cc. of hot HNO_3 (1:1) into a beaker for electrolyzing, and wash the filter thoroughly with hot water. Boil to expel oxides of nitrogen, add 6 to 8 cc. of H_2SO_4 (1:1), dilute to about 150 cc. and electrolyze. Use a weighed sand-blasted cathode and a current of from 3 to 5 amperes at approximately 10 volts. When the solution is colorless and 1 cc. of the solution

[1] 1924 Book of A.S.T.M. Standards.

gives no copper test with H_2S water on a porcelain plate, remove the solution from the electrodes quickly without interrupting the current. Quickly rinse the cathode in distilled water and then dip it in two successive baths of alcohol. Shake off the excess alcohol and ignite the remainder by bringing it to the flame of an alcohol lamp. Keep the cathode moving continually while the alcohol burns. Weigh as metallic copper.

NOTES

The electrolytic method is preferred for the determination of copper no matter what the percentage.

It is not anticipated that the aluminum will contain tin. In case tin has been found it will be preferable to precipitate with hydrogen sulfide. The sulfides are then to be dissolved, digested in HNO_3 (1 : 1), the metastannic acid filtered off and the electrolysis then carried out.

A staining of the anode indicates the presence of lead or manganese.

Copper, when present in small amounts, may also be determined by potassium cyanide titration. See Standard Methods of Chemical Analysis of Alloys of Lead, Tin, Antimony and Copper (Serial Designation: B 18) of the American Society for Testing Materials.[1]

DETERMINATION OF MANGANESE.

(*a*) *Persulfate Method.* (For samples containing under 1.5 per cent of Manganese.)

SOLUTIONS REQUIRED.

"*Acid Mixture.*"—Pour 400 cc. of H_2SO_4 (sp. gr. 1.84) into distilled water, cool and add 400 cc. of HNO_3 (sp. gr. 1.42). Then dilute the mixture with distilled water to a total volume of 2000 cc.

Silver Nitrate.—Dissolve 1.33 g. of $AgNO_3$ in 1000 cc. of distilled water.

Stock Sodium Arsenite Solution.—To 15 g. of arsenious oxide (As_2O_3) in a 300-cc. Erlenmeyer flask, add 45 g. of Na_2CO_3 and 150 cc. of distilled water. Heat the flask and contents on the steam bath until the As_2O_3 is dissolved. Cool the solution, filter and make up to 1000 cc. with distilled water.

Standard Sodium Arsenite Solution.—Mix 200 cc. of the stock sodium arsenite solution with 2500 cc. of distilled water, and standardize by the method described below against a steel or iron of known manganese content. One cubic centimeter of this solution should be equivalent to approximately 0.00050 g. of manganese.

Ammonium Persulfate.—Dissolve 6 g. of ammonium persulfate in 100 cc. of distilled water. (This solution should be made up as needed as it deteriorates rapidly.)

[1] 1924 Book of A.S.T.M. Standards.

METHOD.

In a 250-cc. Erlenmeyer flask, dissolve 1 g. of the sample in 30 cc. of acid mixture by warming on a water bath until solution is complete. Boil until oxides of nitrogen are expelled. Dilute to 100 cc. with boiling hot water, remove the flask from the flame, add 20 cc. of $AgNO_3$ solution, and then 30 cc. of the persulfate solution. Let the solution stand on the steam bath until it has developed a full permanganate color and no bubbles can be seen to come off when the flask is given a whirling motion. Cool to below 25° C., and titrate with the standard arsenite solution to the disappearance of the pink color.

NOTES.

Large amounts (above 0.01 g. of manganese per 100 cc. of solution) of permanganic acid are unstable; the method can, however, be used with satisfaction for alloys containing high percentages of manganese by decreasing the size of the sample.

The ammonium persulfate must be tested for its strength. A good grade should contain about 95 per cent of $(NH_4)_2S_2O_8$; some lots contain no more than 25 per cent of the salt.

If it is desired, larger amounts of the sample can be used and correspondingly greater amounts of $AgNO_3$, NaCl and $(NH_4)_2S_2O_8$ (preferably in the form of stronger solutions).

(*b*) *Bismuthate Method.* (For samples containing less than 1.50 per cent manganese.) (Optional.)

SOLUTIONS REQUIRED.

Dilute Sulfuric Acid (1 : 1).—Pour 500 cc. of H_2SO_4 (sp. gr. 1.84) into distilled water, cool and dilute to 1000 cc.

Nitric Acid (1 : 3).—Pour 250 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 1000 cc.

Dilute Nitric Acid (3-per-cent).—Pour 30 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 1000 cc.

Standard Permanganate Solution.—Dissolve 1 g. of $KMnO_4$ in 1000 cc. of distilled water. Allow it to stand for at least one week and then filter through purified asbestos. Standardize against 0.1 g. portions of pure sodium oxalate. One cubic centimeter of this solution should be equal to approximately 0.00035 g. of manganese.

Standard Ferrous Ammonium Sulfate Solution.—Dissolve 12.4 g. of ferrous ammonium sulfate crystals in 950 cc. distilled water, and add 50 cc. of H_2SO_4 (sp. gr. 1.84).

METHOD.

In a 250-cc. Erlenmeyer flask dissolve 0.5 g. of the sample in 10 cc. of H_2SO_4 (1 : 1), adding a drop or two of HNO_3 (sp. gr. 1.42) occasionally.

When solution is complete heat to expel all oxides of nitrogen and cool. Dilute with 50 cc. of HNO_3 (1 : 3) and add 0.5 g. sodium bismuthate. Heat for a few minutes until the purple color has disappeared with or without the precipitation of manganese dioxide. Add a little ferrous ammonium sulfate solution until the solution becomes clear and boil until the oxides of nitrogen are expelled. Cool, add an excess of sodium bismuthate and agitate for a few minutes. Add 50 cc. of 3-per-cent HNO_3 and filter through an alundum crucible or an asbestos pad. Wash with 50 cc. of 3-per-cent HNO_3. Add from a pipette or a burette 10 to 50 cc. (depending on the amount of permanganic acid) of ferrous ammonium sulfate solution and titrate with the $KMnO_4$ solution.

In exactly the same manner carry through a blank determination using the same amounts of H_2SO_4, HNO_3, and sodium bismuthate as was done with the regular sample. Finally add the exact volume of ferrous ammonium sulfate solution employed and titrate with the $KMnO_4$ solution.

The difference between the volumes required in the two titrations represents the manganese in the sample, and the percentage is found by multiplying this volume by 200 times the manganese titre of the permanganate solution.

NOTES.

Large amounts (above 0.01 g. of manganese per 100 cc. of solution) of permanganic acid are unstable; the method can be used with satisfaction for alloys containing high percentages of manganese by decreasing the size of the sample.

The filtrate from the bismuthate must be perfectly clear, as the least particle of bismuthate carried into the filtrate will vitiate the results.

The solution must be cold at time of filtration and titration.

Instead of employing the method of reducing permanganic acid by means of standardized ferrous ammonium sulfate solution and titrating the excess of the reagent, it is possible to reduce the permanganic acid by standard sodium-arsenite solution. See "Determination of Manganese by the Persulfate Method."

The sodium bismuthate reagent should be tested for manganese and used only if its absence is shown.

ANALYSIS OF LIGHT ALUMINUM ALLOYS.

DETERMINATION OF SILICON, TITANIUM AND IRON.

DETERMINATION OF TOTAL SILICON.

Proceed as described under "Determination of Silicon" in Aluminum.

NOTE.

In case lead sulfate is present (as evidenced by a white crystallin precipitate quickly settling after stirring to the bottom of the casserole) the filtered precipitate

must be washed free from the sulfate with an $HN_4C_2H_3O_2$ wash solution made up as follows: Take 20 cc. NH_4OH (sp. gr. 0.90) add 20 cc. of distilled water and make slightly acid with glacial acetic acid. The final washing must be made with hot water.

DETERMINATION OF TITANIUM.

(*a*) *In the absence of nickel.*

METHOD.

Proceed as described under "Determination of Titanium" in Aluminum.

NOTE.

Vanadium, which interferes in the test, should not be encountered in this class of material. Its presence would be betrayed by an "off" reddish-brown cast in the peroxidized solution.

(*b*) *In the presence of nickel.*

METHOD.

Dissolve 1 g. of the sample as described under "Determination of Tin and Antimony" below. If the nitric acid solution of the sodium hydroxide insoluble is not clear it must be filtered, the residue burned in platinum, fused with a little $K_2S_2O_7$, dissolved in a little 5-per-cent H_2SO_4 and added to the nitric acid filtrate. Dilute the nitric acid solution to 200 cc., treat with NH_4OH (sp. gr. 0.90) until ammoniacal, boil 3 minutes, filter and wash. Dissolve the precipitate in hot dilute H_2SO_4 (25-per-cent) keeping the volume below 80 cc., cool and proceed with the colorimetric test as described under "Determination of Titanium" in Aluminum.

NOTES.

The above method provides for the color interference of copper and nickel when they are present in large amounts.

Vanadium (see Notes above) and other elements which would cause color interferences are not apt to occur in this class of material.

DETERMINATION OF IRON.

SOLUTIONS REQUIRED.

Potassium Permanganate Solution (10-per-cent).—Dissolve 10 g. of $KMnO_4$ in distilled water and dilute to 100 cc.

Dilute Sulfuric Acid for Reductor.—Mix 50 cc. of H_2SO_4 (sp. gr. 1.84) and 1000 cc. of distilled water.

Standard Potassium Permanganate Solution.—Dissolve 1 g. of $KMnO_4$ in 1000 cc. H_2O, and allow to stand for several days in a glass-

stoppered bottle in a dark closet. Filter the solution through prepared asbestos and standardize against 0.1000-g. portions of pure sodium oxalate. Each cubic centimeter is equivalent to approximately 0.0018 g. of iron.

METHOD.

See "Determination of Titanium."

Transfer the solution used in the "Determination of Titanium" from the Nessler tube to a 400-cc. beaker, add 10-per-cent $KMnO_4$ solution to a good permanent pink tint and pass the solution through the reductor. Wash the beaker and reductor with 150 cc. of the H_2SO_4 for reduction and then with 100 cc. of distilled water. Titrate with the standard $KMnO_4$ solution.

The results of the titration should be corrected by a "blank" determination made on 100 cc. of a 7-per-cent H_2SO_4 solution which has been carried through as in the titanium and iron determinations, not omitting the treatment with iron-free zinc, hydrogen peroxide, and permanganate preliminary to the actual reduction. About 0.5 cc. of the permanganate solution will be required to give a permanent color to the blank.

Calculate the apparent percentage of iron. To obtain the true percentage, multiply the percentage of titanium by 1.161 and subtract.

NOTES.

For description and further details of use of reductor see "Chemical Analysis of Iron," by A. A. Blair, or "Quantitative Chemical Analysis," by H. P. Talbot.

Air should never be allowed to enter the body of the reductor.

Titanium is reduced to the trivalent condition in the reduction process and afterwards oxidized to the quadrivalent condition by the permanganate. There is a slight tendency toward oxidation in the receiver, but this may be disregarded in the small amounts of titanium ordinarily involved.

In case titanium is present and iron alone is to be determined proceed as follows: Place the H_2SO_4 solution of the sample in a 250-cc. flask, dilute to 100 cc. containing 2.5 cc. H_2SO_4 (sp. gr. 1.84) and treat with H_2S for 30 minutes in the cold and 15 minutes while warmed. If sulfides appear, filter and gas the filtrate 10 minutes more. Add 15 cc. H_2SO_4 (1 : 1) and boil vigorously until H_2S is expelled and the volume reduced to 50 cc. Fill the flask with distilled water, transfer to a beaker, and titrate with $KMnO_4$ solution.

In case the alloy contains high percentages of copper, lead, tin or antimony it is preferable to treat the combined filtrate and washings from the silicon determination with H_2S and filter off and wash the sulfates. The solution should then be boiled free from hydrogen sulfide, treated with 10-per-cent $KMnO_4$ solution to a pink tint and then reduced as directed.

Reducible elements other than iron and titanium must be absent. Such elements are chromium, vanadium, molybdenum, tungsten and uranium, and they ordinarily will not be found in this class of material.

DETERMINATION OF ANTIMONY, TIN, LEAD AND COPPER.

PRELIMINARY SEPARATION.

SOLUTIONS REQUIRED.

Sodium Hydroxide Solution (25-per-cent).—Dissolve 250 g. of NaOH in distilled water and dilute to 1000 cc. If a sediment forms on standing, decant the solution and filter through an asbestos pad. The NaOH must be entirely free from organic matter.

Nitric Acid (1 : 1).—Pour 500 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 1000 cc.

METHOD.

Dissolve 5 g. of the sample in a covered porcelain dish of suitable size in 125 cc. of (25-per-cent) NaOH solution. When solution is complete dilute with 100 cc. of distilled water, decant off the clear solution and wash the residue well with hot dilute NaOH solution (2-per-cent). Treat the filtrate and washings with H_2S. If no precipitate appears, zinc is absent. In case a precipitate appears, zinc must be determined in a fresh sample as described further. Unfold the wet paper containing the NaOH insoluble and wash the residue back into the original vessel. Replace the paper in the funnel and pour 20 cc. of hot HNO_3 (1 : 1) over the paper and allow it to filter into the original vessel. Boil the residue until action ceases and oxides of nitrogen are expelled. Add 50 cc. of hot distilled water and allow to stand and settle for about one hour, keeping the temperature just below the boiling point. Filter on an ashless paper, being careful to keep the solution hot throughout the process of filtration, and reclev-ing the filtrate in a 200-cc. beaker of the tall type used in electrolysis. Wash with boiling hot water. Retain the filtrate and washings for subsequent use in the copper and lead determinations and treat the residue as described under "Determination of Antimony."

NOTES.

Antimony will be quantitatively recovered with the tin provided it does not exceed one-fifth its amount. In the rare case of high antimony evaporate the nitric acid solution of the NaOH insoluble to dryness, bake at 105° C., take up in hot 10-per-cent by volume nitric acid, and proceed with the filtration and washing.

The metastannic acid precipitate will not contain enough copper to make a recovery worth while in ordinary work. As iron is determined in a separate portion and does not interfere in the determinations of antimony and tin, it may also be disregarded.

DETERMINATION OF ANTIMONY

SOLUTIONS REQUIRED.

Standard Permanganate Solution.—The solution prepared as under "Determination of Iron" may be employed. It must be

standardized against weighed 0.05-g. portions of pure antimony or suitable material of known antimony content carried through the regular procedure after solution in acid and treatment in the Kjeldahl flask.

METHOD.

See "Preliminary Separation."

Transfer the filter containing metastannic acid and antimony oxide to a 500-cc. Kjeldahl flask, add 10 to 12 cc. H_2SO_4 (sp. gr. 1.84) and 3 to 5 g. Na_2SO_4. Heat until all organic matter is decomposed. Dilute, when cool, with 20 cc. of water. Add 0.5 g. Na_2SO_3 and boil until all SO_2 is expelled from the flask. Dilute to about 250 cc., add 20 cc. of HCl (sp. gr. 1.20) cool to 10° C. and titrate with the $KMnO_4$ solution to the first definite shade of pink. Reserve the solution for the "Determination of Tin."

DETERMINATION OF TIN.

SOLUTIONS REQUIRED.

Standard Iodine Solution.—Dissolve 10.7 g. of iodine in 50 cc. of water containing 20 g. of KI in solution, and dilute to 1000 cc. *when the iodine is completely in solution.* Standardize against pure tin by the method described below.

Starch Solution.—Prepare as described under "Determination of Copper, Iodide Method."

METHOD.

See "Determination of Antimony."

Transfer the solution used in "Determination of Antimony" to a 450-cc. Erlenmeyer flask, add an excess of powdered antimony and 50 cc. of HCl (sp. gr. 1.20). Attach a reflux condenser and boil for 15 minutes on a hot plate. Cool in a current of CO_2, add 5 cc. of starch solution, and without disconnecting the stream of CO_2, titrate with the iodine solution to the blue end point.

DETERMINATION OF COPPER AND LEAD SIMULTANEOUSLY. BY THE ELECTROLYTIC METHOD.

APPARATUS REQUIRED.

See "Determination of Copper" under "Analysis of Aluminum."

METHOD.

See "Preliminary Separation."

Dilute the filtrate and washings from the "Preliminary Separa-

tion" to about 150 cc. with distilled water and electrolyze, using weighed sand-blasted cathode and anode. Cover with a pair of split watch glasses and use a current of from 1.25 to 1.5 amperes, at approximately 10 volts for each solution. After about one hour the lead will have been entirely deposited on the anode as PbO_2; without interrupting the current add to the electrolyte 3 to 4 cc. of H_2SO_4 (sp. gr. 1.84), wash down the cover glasses, electrodes and sides of the beaker and continue the electrolysis for 15 minutes. If no darkening of the newly-exposed anode surface can be detected and if a few drops of H_2S water added to 1 cc. of the electrolyte on a porcelain plate gives no coloration the electrolysis is complete. In this case, remove the solution from the electrodes quickly without interrupting the current. Rinse the electrodes in quick succession with distilled water and then twice with alcohol. Reserve the electrolyte and water washings for the nickel and magnesium determinations. Dry the anode at 210° C. for thirty minutes. Weigh as PbO_2, using the factor for lead 0.8643 instead of the theoretical value. Shake off the excess alcohol from the cathode and ignite the remainder by bringing it to the flame of an alcohol lamp. Keep the cathode moving continually while the alcohol burns. Weigh as metallic copper.

NOTE.

The electrolyte and water washings may be diluted to 500 cc. and one 250-cc. portion taken for the "Determination of Nickel" and the other for the "Determination of Magnesium" as directed further. If the alloy contains copper and no tin, antimony or lead the electrolytic method given under "Determination of Copper" in Aluminum may be employed.

DETERMINATION OF LEAD AS SULFATE.

(OPTIONAL.)

SOLUTIONS REQUIRED.

"*Lead Acid.*"—Mix 300 cc. of H_2SO_4 (sp. gr. 1.84) and 1800 cc. of distilled water. Dissolve 1 g. of lead acetate C.P. in 300 cc. of distilled water and add this to the hot solution, stirring meanwhile. Let stand at least 24 hours and siphon through a thick asbestos filter.

Dilute Alcohol for Washing.—Mix equal parts of denatured alcohol and distilled water.

METHOD.

See "Preliminary Separation."

To the combined filtrate and washings from the "Preliminary Separation" add 120 cc. of lead acid, and evaporate until copious fumes of H_2SO_4 are evolved. Cool, add 105 cc. of distilled water to dissolve the salts and to make the concentration the same as in the lead acid,

heat to boiling, and allow to cool and settle for 5 hours, or over night if convenient. Filter on a weighed porcelain Gooch crucible and wash with lead acid. Set the filtrate and washings aside for the "Determination of Copper by the Iodide Method." Continue the washing and wash out the lead acid with dilute alcohol. Set the Gooch crucible inside a porcelain crucible, dry and ignite for 5 minutes at the full heat of a Tirrill burner. Cool and weigh as $PbSO_4$, which contains 68.32 per cent lead.

DETERMINATION OF NICKEL BY THE DIMETHYLGLYOXIME METHOD.

SOLUTIONS REQUIRED.

Sulfuric Acid (1 : 1).—Pour 500 cc. of H_2SO_4 (sp. gr. 1.84) into distilled water, cool, and dilute to 1000 cc.

Hydrogen Sulfide Wash Water.—Add 10 cc. of H_2SO_4 (sp. gr. 1.84) to 1000 cc. of distilled water and saturate with H_2S.

Tartaric Acid Solution (20-per-cent).—Dissolve 20 g. of tartaric acid in distilled water, dilute to 100 cc. and filter if necessary.

Dimethylglyoxime Solution (1-per-cent).—Dissolve 10 g. of the reagent in 1000 cc. of 95-per-cent alcohol.

METHOD.

Dissolve 1 g. of the sample in 25 cc. of H_2SO_4 (1 : 1) as directed in "Determination of Manganese by the Persulfate Method." After oxides of nitrogen have been expelled add 14 cc. NH_4OH in order to bring down the acidity to approximately 5 cc. H_2SO_4 (sp. gr. 1.84) per 100 cc. of solution. Treat with hydrogen sulfide. Filter off the sulfides, wash with hydrogen sulfide wash water and boil the filtrate until H_2S is expelled. Add a few crystals of $(NH_4)_2S_2O_8$ and boil thoroughly to destroy sulfur and oxidize iron. Add 20 cc. of tartaric acid solution and make slightly ammoniacal. If no precipitate appears add HCl (sp. gr. 1.20) to slight acidity. If a precipitate appears, dissolve in HCl add 10 cc. of tartaric acid and again make ammoniacal, repeating the operation, if necessary, to get a clear solution. To the weakly acid solution add dimethylglyoxime in such an amount that the ratio of the reagent to nickel is at least 4 : 1. Heat the solution to boiling, and make slightly ammoniacal. Allow to digest while cooling for two hours. Collect on a weighed Gooch crucible and wash thoroughly with hot water. Dry for forty-five minutes at 110 to 120° C. and weigh. The nickel dimethylglyoxime contains 20.32 per cent of nickel.

NOTES.

In case copper and lead have been determined electrolytically, nickel may be determined in one 250-cc. portion of the electrolyte and washings (see Notes), starting with the $(NH_4)_2S_2O_8$ treatment to oxidize iron and then proceeding as directed in the method above.

Nickel may be precipitated by glyoxime, filtered, dissolved in nitric acid, made ammoniacal and titrated with cyanide solution according to Frevert's method.[1]

DETERMINATION OF MAGNESIUM.

SOLUTIONS REQUIRED.

Dilute Hydrochloric Acid (1 : 1).—Pour 500 cc. of HCl (sp. gr. 1.20) into distilled water and dilute to 1000 cc.

Sulfuric Acid Wash Water.—Saturate 500 cc. of a 1-per-cent H_2SO_4 solution with H_2S.

Ammonium Sulfide Wash.—Take 10 cc. of NH_4OH, dilute to 500 cc., add 10 g. NH_4Cl and saturate with H_2S.

Microcosmic Salt Solution.—Prepare a clear saturated solution as required.

Ammoniacal Wash Solution.—Dissolve 50 g. of NH_4NO_3 in distilled water, add 50 cc. NH_4OH (sp. gr. 0.90) dilute to 1000 cc. and filter if necessary.

METHOD.

Dissolve 10 g. of the sample and wash the residue as directed under the "Preliminary Separation Determination of Antimony, Tin, Lead and Copper." Dissolve the residue in 10 cc. hot HCl (1 : 1) using a little HNO_3 if necessary. If the hydrogen sulfide group of metals are present, neutralize with NH_4OH, make weakly acid with HCl, dilute to 100 cc., heat and precipitate with hydrogen sulfide. Filter off the sulfides and wash with the H_2SO_4 wash water. To this filtrate or to the HCl solution if no H_2S treatment was required, add 5 cc. HCl (sp. gr. 1.20) make slightly ammoniacal and precipitate with H_2S. Let the solution stand for about two hours at a temperature of approximately 40° C. (at the side of the steam bath). Filter and wash with the $(NH_4)_2S$ wash. Acidify the filtrate with HCl, boil off H_2S, add $(NH_4)_2S_2O_8$ and boil to destroy sulfur if necessary. Add 1 g. of tartaric acid and 30 cc. of microcosmic salt solution. Cool, and add NH_4OH (sp. gr. 0.90) drop by drop while stirring vigorously until a crystallin precipitate begins to form. Continue the very slow addition of NH_4OH with stirring until a precipitate no longer forms and then add one-tenth the volume of NH_4OH (sp. gr. 0.90) and let stand over night. Filter and wash with cold ammoniacal

[1] A. A. Blair, "The Chemical Analysis of Iron," 8th Edition, Lippincott and Co., pp. 182–183.

wash solution. Ignite slowly under oxidizing conditions in a tared porcelain or platinum crucible and finally heat over the blast lamp or its equivalent. Weigh as $Mg_2P_2O_7$. The weight multiplied by 0.2185 represents magnesium.

Note.

In case copper and lead were determined electrolytically one reserved portion of the electrolyte and washings (see Notes) may be used for the determination of magnesium, starting in with the addition of 5 cc. HCl (sp. gr. 1.20) and the precipitation of the $(NH_4)_2S$ group.

DETERMINATION OF MANGANESE.

(*a*) *Persulfate or Bismuthate Method.* (For samples containing under 1.50 per cent manganese.)

Proceed as directed in "Determination of Manganese" under "Analysis of Aluminum."

(*b*) *Nitric Acid and Potassium Chlorate Method.*[1] (For samples containing over 1.50 per cent manganese.)

Solutions Required.

Hydrochloric Acid (1 : 1).—Mix 500 cc. of HCl (sp. gr. 1.20) and 500 cc. of distilled water.

Sulfurous Acid.—Pass SO_2 from a cylinder into 500 cc. of cold distilled water until saturated.

Bromine Water.—Place 20 to 30 cc. of liquid bromine in a 500-cc. bottle, fill with cold distilled water, and shake thoroughly. *Avoid any contact with the skin.*

Dilute Hydrochloric Acid (1 : 3.)—Pour 250 cc. of HCl (sp. gr. 1.20) into distilled water and dilute to 1000 cc.

Sodium-Ammonium Phosphate Solution.—Prepare this solution as needed by dissolving 1 part of the salt in 6 parts of cold water and filtering.

Ammonium Nitrate Solution.—Dissolve 100 g. of NH_4NO_3 in distilled water, make slightly ammoniacal, dilute to 1000 cc. and filter if necessary.

Method.

In a 250-cc. Erlenmeyer flask dissolve 1 g. of the sample in 30 cc. of. HCl (1 : 1). When solution is complete add 25 cc. HNO_3 (sp. gr. 1.42) and evaporate until the solution is almost syrupy. Add 50 cc. of HNO_3 (sp. gr. 1.42) and 5 g. $KClO_3$. Heat the solution to boiling either on the hot plate or on a tripod with a thin piece of sheet asbestos, about 1 in. (25 mm.) in diameter, on the center of the wire gauze.

[1] A. A. Blair, "The Chemical Analysis of Iron," 8th Edition, J. B. Lippincott and Co., pp. 108–115.

Boil the solution 15 minutes, remove from the heat, add 50 cc. HNO_3 (sp. gr. 1.42) and 5 g. $KClO_3$ and boil 15 minutes longer or until yellowish fumes are no longer given off. Cool the solution rapidly in cold water and filter through asbestos. Wash two or three times with HNO_3 (sp. gr. 1.42) (free from oxides of nitrogen which dissolve the dioxide).

Transfer the dioxide with the asbestos filter to the beaker in which the precipitation was made. Pour from 10 to 40 cc. of strong sulfurous acid through the filtering apparatus into the beaker. As soon as the precipitate has dissolved, filter from the asbestos into a 150-cc. beaker, wash with hot water, and add 5 cc. HCl (sp. gr. 1.20). Heat to expulsion of SO_2, add bromine water until the solution is strongly colored with it, and boil off all excess bromine. Add NH_4OH in slight excess, boil for a few minutes, and filter into a 600-cc. beaker. Wash with hot water, remove and reserve the filtrate, and dissolve the precipitate in dilute HCl (1 : 3), allowing the solution to run back into the beaker in which the precipitation was made. Wash the filter with hot water. Boil the solution for 10 minutes to drive off any chlorine and reprecipitate by NH_4OH as before, filter into the reserved filtrate and repeat the solution, precipitation and filtration, allowing all the filtrates from the $Fe(OH)_3$ to run into the 600-cc. beaker. Acidulate this solution, which will be about 300 or 400 cc. in volume, with acetic acid, heat to boiling and treat with H_2S for 10 minutes. Filter, wash with a little dilute acetic acid saturated with hydrogen sulfide and boil off the hydrogen sulfide after adding 5 cc. of HCl (sp. gr. 1.20). Add from 5 to 20 cc. of a clear filtered solution of microcosmic salt, heat to boiling, and add NH_4OH drop by drop with constant stirring. When the precipitate begins to form, stop the addition of NH_4OH and stir until the precipitate becomes crystallin. When this change occurs add one more drop of ammonia; the additional precipitate formed will be curdy, but a few seconds stirring will change it to the silky crystallin condition. Continue the addition of NH_4OH in exactly the same manner until the precipitate is all down and further additions of NH_4OH fail to change the silky appearance. Add a dozen drops of NH_4OH in excess and cool the beaker in ice water. Filter the precipitate on an asbestos filter, wash with the cold NH_4NO_3 solution until the filtrate gives no reaction for HCl. Dry, ignite and weigh as $Mn_2P_2O_7$ which contains 38.69 per cent manganese.

NOTES.

The Ford method is recommended only where extreme accuracy is desired, otherwise use the persulfate or bismuthate methods.

If a cylinder of SO_2 is not available, hydrogen peroxide will serve equally well for the solution of the hydrated manganese dioxide.

DETERMINATION OF ZINC.

(*a*) *Samples containing up to 3 per cent of zinc.*

SOLUTIONS REQUIRED.

Dilute Hydrochloric Acid (1 : 1).—Pour 500 cc. of HCl (sp. gr. 1.20) into distilled water and dilute to 1000 cc.

Acid Wash Water.—Pour 10 cc. of H_2SO_4 (sp. gr. 1.84) into 1000 cc. of distilled water and saturate with H_2S.

Formic Acid Mixture.—Treat 400 cc. of formic acid with 60 cc. of NH_4OH (sp. gr. 0.90) and dilute to 2000 cc.

Formic Acid Wash Water.—Pour 25 cc. of formic acid mixture into 1000 cc. of distilled water and saturate with H_2S.

Dilute Hydrochloric Acid (1 : 10).—Pour 100 cc. of HCl (sp. gr. 1.20) into distilled water and dilute to 1000 cc.

Bromine Water.—See "Determination of Manganese, Nitric Acid and Potassium Chlorate Method."

METHOD.

Dissolve 1 g. of the sample in a covered 400-cc. beaker in 25 cc. of dilute HCl (1 : 1), with the addition of 1 cc. of HNO_3 (sp. gr. 1.42) toward the end of the reaction. Boil to expel oxides of nitrogen and dilute to 200 cc. Precipitate the H_2S group, filter and wash with acid wash water. Boil thoroughly to expel H_2S, cool, and add 5 g. of citric acid crystals. Neutralize with NH_4OH, using methyl orange as an indicator and then add 25 cc. of the formic acid mixture. Dilute to 300 cc., heat nearly to boiling and pass in H_2S until the solution is cold. Filter and wash with the formic acid wash water. Dissolve the zinc sulfide off the filter with warm dilute HCl (1 : 10) and wash the filter with hot water. Boil the solution to expel H_2S. Cool, add 1 g. of citric acid crystals, neutralize with NH_4OH, using methyl orange as an indicator and then add 20 cc. of the formic acid mixture. Dilute to 200 cc. and proceed as before through the filtration on an ashless paper and washing of the sulfide. Dissolve the precipitate with hot dilute HCl, and transfer the solution to a weighed platinum or porcelain dish or crucible. Add a few drops of H_2SO_4 (sp. gr. 1.84) and evaporate the solution until copious fumes escape. If the solution is not clear and colorless, cool, add a few cubic centimeters of HNO_3 (sp. gr. 1.42) and again evaporate the solution until fumes of H_2SO_4 come off freely. Repeat the treatment with HNO_3 if necessary, until the organic matter is destroyed, and the solution is colorless. Remove the excess of H_2SO_4 by heating the dish cautiously, and finally heat at

a temperature just below dull redness. Weigh as $ZnSO_4$, which contains 40.49 per cent of zinc.

NOTE.

The use of a sodium hydroxide solution of the turnings is not recommended for the determination of zinc on account of uncertainty as to the completeness of the solution of the zinc in all cases.

(*b*) *Samples containing over 3 per cent of zinc.*[1]

SOLUTIONS REQUIRED.

Ferrous Sulfate Solution.—Dissolve 3 g. of $FeSO_4 . 7H_2O$ in distilled water, add 10 cc. of HCl (sp. gr. 1.20) and dilute to 1000 cc.

Ferrocyanide Solution.—Dissolve 44 g. of K_4FeCN_6 and 0.3 g. of K_3FeCN_6 in distilled water, dilute to 1000 cc. and age six weeks before standardization as follows: Dissolve 6 g. of zinc of known content in 40 cc. of HCl (sp. gr. 1.20) in a 2-liter flask. Cool and make up to the mark. Take 100-cc. portions, add 20 cc. of HCl (1 : 1) and add 13 cc. of NH_4OH (sp. gr. 0.90) and proceed with the titration as directed below.

METHOD.

Proceed as in the previous method until the first zinc sulfide precipitate has been obtained. Return the paper and precipitate to the original beaker and add 20 cc. of HCl (1 : 1). When the sulfide has dissolved, filter off the paper together with some sulfur and possibly a little nickel sulfide. Boil the solution containing the zinc to expel H_2S, cool, and wash down. Add 13 cc. of NH_4OH (sp. gr. 0.90) and if the solution is not alkaline, add NH_4OH carefully until it is. Make the solution barely acid again with HCl (1 : 1) and add 3 cc. of HCl (sp. gr. 1.20) in excess. Add 1 cc. of $FeSO_4$ solution, dilute nearly to 200 cc., heat almost to boiling and titrate with the K_4FeCN_6 solution. The end point is a sharp change in the color of the solution from a turquoise blue to a "pea green" and with several more drops to a "creamy yellow."

NOTE.

The end point occurs a little sooner than the one with uranium nitrate, and is easier to use, as the change is seen directly in the solution.

DETERMINATION OF SODIUM.

SOLUTIONS REQUIRED.

Dilute Nitric Acid (1 : 2).—Pour 250 cc. of HNO_3 (sp. gr. 1.42) into distilled water and dilute to 750 cc.

[1] Method of F. G. Breyer, 8th International Congress of Applied Chemistry, 1, 162; see also "Analysis of Copper," G. L. Heath, McGraw-Hill Book Co., pp. 250, 251.

Saturated Ammonium Carbonate Solution.—Small quantities made up as required.

Method.

Dissolve 5 g. of the sample in a porcelain dish in 100 cc. of dilute HNO_3 (1 : 2) by continued digestion at 50 to 60° C. When solution is complete, evaporate the solution to dryness and heat on a sand bath for several hours to complete decomposition of aluminum nitrate, but without melting any sodium nitrate formed. Cool the residue, take it up in boiling distilled water, filter and wash thoroughly. Treat the water extract with saturated $(NH_4)_2CO_3$ solution, heat to boiling and filter if a precipitate forms. Evaporate the clear filtrate in a weighed platinum dish, treat the residue with sufficient H_2SO_4 to convert the nitrate to sulfate, continue the evaporation and finally heat at 300 to 400° C. to complete expulsion of H_2SO_4. Weigh as Na_2SO_4. A blank should be carried through all operations and the proper correction applied.

DETERMINATION OF NITROGEN.

Solutions Required.

Sodium Hydroxide Solution (10-per-cent).—Dissolve 100 g. of NaOH in distilled water, dilute to 1000 cc. and filter through asbestos if the solution develops a sediment upon standing.

Approximate N/10 Sulfuric Acid Solution.—Pour 2.8 cc. of H_2SO_4 (sp. gr. 1.84) into 1000 cc. of distilled water and standardize by any standard method.

Approximate N/10 Sodium Hydroxide Solution.—Dissolve 4.5 g. of NaOH in distilled water and dilute to 1000 cc. This solution should be standardized against the standard acid solution.

Method.

Place 3 to 4 g. of the sample in a Kjeldahl flask fitted with a two-hole stopper carrying a separatory funnel and a trap connected with a condenser as in a Kjeldahl determination of nitrogen. Add 400 cc. of the NaOH solution through the separatory funnel, and allow the generated gas to bubble through 50 cc. of N/10 sulfuric acid solution. When the reaction has ceased, boil the alkaline solution to expel all ammonia. Finally titrate the acid with N/10 sodium hydroxide in the usual manner and calculate nitrogen.

DETERMINATION OF ALUMINUM AND ALUMINUM OXIDE.

SOLUTIONS REQUIRED.

Sodium Hydroxide Solution (10-per-cent).—Dissolve 100 g. of NaOH in distilled water and dilute to 1000 cc. If a sediment forms on standing, decant the solution and filter through an asbestos pad.

Sodium Carbonate Solution.—Dissolve 36 g. of Na_2CO_3 in distilled water, dilute to 1000 cc., and filter if necessary.

Barium Chloride Solution.—Dissolve 90 g. of $BaCl_2.2H_2O$ in distilled water, dilute to 1000 cc., and filter if necessary.

Dilute Hydrochloric Acid.—Dilute 500 cc. of HCl (sp. gr. 1.20) with 500 cc. of water.

Methyl Red Indicator.—Dissolve 0.02 g. of the acid in 100 cc. of distilled water.

Ammonium Chloride Wash Solution.—Dissolve 50 g. of NH_4Cl in distilled water, make sure the solution is just alkaline to methyl red, dilute to 1000 cc. and filter if necessary.

Dilute Nitric Acid (1 : 1).—Dilute 500 cc. of HNO_3 (sp. gr. 1.42) with 500 cc. of distilled water.

Acid Wash Water.—Add 5 cc. of H_2SO_4 (sp. gr. 1.84) to 500 cc. of water and saturate with H_2S.

PRELIMINARY TREATMENT.

Place 2 g. of the sample in a suitable porcelain dish, add 60 cc. of the NaOH solution (10-per-cent), cover with a glass, and cool in water if the reaction proceeds too violently. When violent action ceases, wash the cover-glass and sides of the vessel, replace the glass and warm on a sand bath or steam-box until reaction ceases. These operations should be carried on expeditiously. Filter and wash the residue with hot NaOH solution (10-per-cent). Reserve the residue and treat the filtrate and washings as below.

(*a*) *Determination of Aluminum.*

Cool the filtrate and washings to room temperature, dilute to exactly 500 cc. and mix thoroughly. Transfer pipetted 50-cc. portions to 600-cc. beakers, add 10 cc. of HCl (sp.gr. 1.20), place on water bath and evaporate to dryness. When dry, continue the heating for one hour more. Take up the residue in cold distilled water, transfer to a flask and add a mixture of 50 cc. of the Na_2CO_3 solution and 50 cc. of the $BaCl_2$ solution. Shake thoroughly and allow to settle. Filter, wash with cold water, and then transfer the precipitate to the original flask by piercing the paper and washing down with boiling water,

finally with dilute HCl, adding enough to dissolve the precipitate and avoiding mechanical loss. Evaporate the solution to one-third volume, add a few drops of methyl red and then NH_4OH (sp. gr. 0.90) until the indicator just turns yellow. Boil for three minutes, filter and wash with warm 5-per-cent NH_4Cl solution. Bring the precipitate back into the beaker as before, dissolving all $Al(OH)_3$ on the paper and evaporate the solution to dryness. Drench with a little HCl (sp. gr. 1.20) add hot water and warm until salts are in solution. Filter in case a residue is present, reprecipitate $Al(OH)_3$ and wash as above. Place the wet paper and contents in a tared crucible (with cover and preferably of platinum), dry, ignite carefully in an oxidizing atmosphere and finally cover and blast strongly. Weigh as Al_2O_3 and calculate aluminum.

NOTES.

This method is a modification of the method proposed by J. G. Rhodin, Faraday Society Trans., *14*, (1914–19) pp. 135–7 and presupposes complete solubility of aluminum and insolubility of aluminum oxide in a 10-per-cent solution of NaOH.

It is important that the "50-cc." pipette deliver exactly one-tenth of the "500-cc." volume.

Two precipitations with NH_4OH are necessary to eliminate barium and the intervening evaporation to dryness is desirable in case silica is present.

(*b*) *Determination of Aluminum Oxide.*

See "Preliminary Separation."

Transfer the residue insoluble in NaOH solution (10-per-cent) to a small flask and treat the paper with hot dilute HNO_3 (1:1). When solution of the residue is complete, dilute to 100 cc., neutralize with NaOH solution (10-per-cent), add 3 cc. HCl (sp. gr. 1.20) and treat with hydrogen sulfide. Filter, wash the sulfides with acid wash water and boil the filtrate and washings until H_2S is expelled, using a few crystals of $K_2S_2O_8$ if sulfur separates. Pour the hot solution slowly and with stirring into 50 cc. of boiling hot NaOH solution (10-per-cent). Filter and wash with a little hot NaOH solution (10-per-cent).

In case the precipitate is large, dissolve it and reprecipitate. Acidify the NaOH filtrate or filtrates with HCl (sp. gr. 1.20) and precipitate $Al(OH)_3$ as described in (*a*) above. A second precipitation should be carried out in case the precipitate is large, and always in accurate work. Ignite, weigh as Al_2O_3 and report as such.

TENTATIVE SPECIFICATIONS AND TESTS

FOR

COMPRESSIVE STRENGTH OF PORTLAND-CEMENT MORTARS.[1]

Serial Designation: C 9 – 16 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1916.

SPECIFICATIONS.

Compressive Strength.

1. The average compressive strength in pounds per square inch of not less than three standard mortar test pieces (see Section 4) composed of one part cement and three parts standard sand, by weight, shall be equal to or higher than the following:

Age at Test, days.	Storage of Test Pieces.	Compressive Strength, lb. per sq. in.
7	1 day in moist air, 6 days in water........................	1200
28	1 day in moist air, 27 days in water........................	2000

Strength at 28 days.

2. The average compressive strength of standard mortar at 28 days shall be higher than the strength at 7 days.

TESTS.

Mixing Standard Mortar.

3. The requirements governing the preparation of standard sand mortars for tension test pieces shall apply to compression test pieces.[2]

[1] Criticisms of these Tentative Specifications and Tests are solicited and should be directed to Mr. R. B. Young, Secretary of Committee C-1 on Cement, Hydro-Electric Power Commission of Ontario, 8 Strachan Ave., Toronto, Ontario, Canada.

These specifications and tests, when adopted as standard, will be added to the present Standard Specifications and Tests for Portland Cement (Serial Designation: C 9–21), 1924 Book of A.S.T.M. Standards.

[2] See Standard Specifications and Tests for Portland Cement (Serial Designation: C 9), 1924 Book of A.S.T.M. Standards.

Form of Test Piece. 4. A cylindrical test piece 2 in. in diameter and 4 in. in length is recommended for use in making compression tests of standards mortars. The molds shall be made of non-corroding metal. A satisfactory form of mold is shown in Fig. 1. The ends of the mold shall be parallel. The tubing used in the molds shall be of sufficient thickness to prevent appreciable distortion. The molds shall be oiled before using. During the

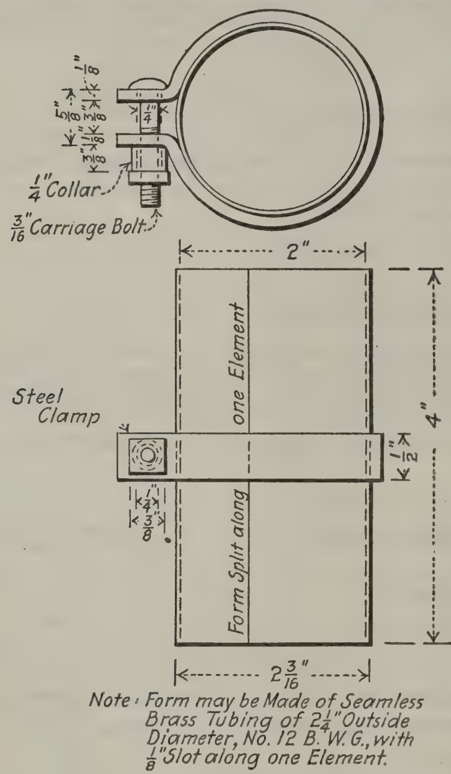

FIG. 1.—Details for 2 by 4-in. Cylinder Form.

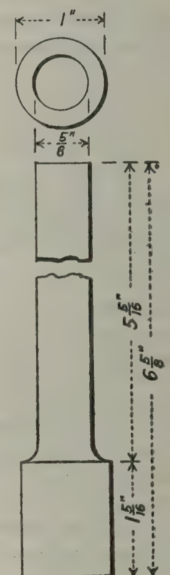

FIG. 2.—Details or Steel Tamper.

molding of the test piece, the mold shall rest on a clean, plane surface (preferably a piece of plate glass which is allowed to remain in place until the mold is removed).

Molding. 5. The mortar[1] shall be placed in the mold in layers about 1 in. in thickness, each layer being tamped by means of the steel tamper shown in Fig. 2. The weight of tamper shall be

[1] If sufficient mortar for six 2 by 4-in. cylinders is to be mixed in a single batch, approximately 3000 g. of material will be required. In this case the mixing shall be continued for 1½ minutes.

approximately ¾ lb. In finishing the test piece, the mortar shall be heaped above the mold and smoothed off with a trowel. As soon as the test pieces from one sample are molded, the top of each test piece shall be covered with a piece of glass which is brought to a firm bearing on the fresh mortar. The cover glasses shall remain in place until the molds are removed.

6. The compression test pieces shall be stored in the same manner as the tension test pieces. **Storage.**

7. Tests of standard-mortar cylinders shall be made in any testing machine which is adapted to meet the specified requirements. The test pieces shall be tested as soon as removed from the water. The ends of the test cylinders shall be smooth, plane surfaces. The metal bearing plates of the testing machine shall be placed in direct contact with the ends of the test piece. During the test a spherical bearing block shall be used on top of the cylinder. In order to secure a uniform distribution of the load over the test cylinder the spherical bearing block must be accurately centered. The diameter of the spherical bearing block should be only a little greater than that of the test piece. The test piece shall be loaded continuously to failure. The moving head of the testing machine shall travel at the rate of not less than 0.05 or more than 0.10 in. per minute. **Testing.**

8. Testing machines should be frequently calibrated in order to determine their accuracy. **Calibration.**

9. Cylinders that are manifestly faulty, or which give strengths differing more than 15 per cent from the average value of all test pieces tested at the same period and made from the same sample, shall not be considered in determining the compressive strength. **Faulty Cylinders.**

TENTATIVE SPECIFICATIONS

FOR

REQUIRED SAFE CRUSHING STRENGTHS OF SEWER PIPE TO CARRY LOADS FROM DITCH FILLING.[1]

Serial Designation: C 15 – 17 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1917.

The following table gives required safe crushing strengths per linear foot of pipe, to carry loads from ditch filling materials only when pipe is laid in accordance with the Recommended Practice for Laying Sewer Pipe (Serial Designation: C 12) of the American Society for Testing Materials,[2] for sand and for thoroughly wet clay ditch filling materials. In the preparation of this table a safety factor of 1½ has been used, which has been found necessary to prevent cracking from the loads of ditch filling.[3]

Ordinary Pipe Laying is pipe laying in accordance with customary good practice in pipe-sewer construction, whereby the under side of the pipe is well bedded on soil for 60 to 90 deg. of the circumference.

First Class Pipe Laying is pipe laying in accordance with the best customary practice in pipe-sewer construction, whereby the entire under side of the pipe is very thoroughly bedded on soil and the entire pipe is surrounded by well-compacted soil,

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. E. S. Rankin, Secretary of Committee C-4 on Clay and Cement-Concrete Sewer Pipe, Bureau of Sewers, City Hall, Newark, N. J.,

[2] 1924 Book of A.S.T.M. Standards.

[3] Prepared from the Standard Specifications for Drain Tile (C 4), 1924 Book of A.S.T.M. Standards.

under the direction of an inspector constantly present on the work.

SAFE CRUSHING STRENGTHS OF SEWER PIPE TO CARRY LOADS FROM DITCH FILLING FOR ORDINARY SAND AND FOR THOROUGHLY WET CLAY DITCH FILLING MATERIALS.
(FOR ORDINARY PIPE-LAYING METHODS.)

STRENGTHS IN POUNDS PER LINEAR FOOT.

Height of Fill above Top of Pipe, ft.	Breadth of Ditch a Little Below Top of Pipe.									
	1 ft.		2 ft.		3 ft.		4 ft.		5 ft.	
	Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.	
	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.
2..........	265	280	615	635	970	990	1 330	1 350	1 690	1 710
4..........	400	450	1 055	1 125	1 745	1 825	2 455	2 535	3 165	3 250
6..........	470	545	1 370	1 500	2 370	2 525	3 405	3 575	4 460	4 640
8..........	505	605	1 600	1 790	2 875	3 115	4 215	4 495	5 595	5 890
10..........	525	640	1 765	2 015	3 275	3 610	4 900	5 295	6 590	7 020
12..........	535	660	1 880	2 185	3 600	4 030	5 485	6 000	7 460	8 085
14..........	540	675	1 965	2 320	3 855	4 380	5 975	6 620	8 225	8 950
16..........	545	680	2 025	2 425	4 065	4 675	6 395	7 165	8 890	9 775
18..........	545	685	2 070	2 505	4 230	4 920	6 750	7 630	9 480	10 520
20..........	545	690	2 100	2 565	4 365	5 130	7 050	8 060	9 995	11 190
22..........	545	690	2 125	2 610	4 470	5 305	7 305	8 425	10 445	11 795
24..........	545	690	2 140	2 645	4 560	5 455	7 525	8 750	10 840	12 340
26..........	545	690	2 150	2 675	4 630	5 575	7 705	9 035	11 185	12 830
28..........	545	690	2 160	2 695	4 685	5 680	7 860	9 280	11 490	13 270
30..........	545	690	2 165	2 715	4 725	5 765	7 990	9 500	11 755	13 670
Very great	545	690	2 180	2 770	4 910	6 230	8 725	11 075	13 635	17 805

When pipe are laid in a *Concrete or other Permanent Masonry Cradle,* strong enough to carry the entire load to the sub-base without breaking and large enough to prevent material settlement, the standard strengths for all dimensions of ditches and all filling materials shall be those specified for standard sewer pipe.

TENTATIVE SPECIFICATIONS

FOR

QUICKLIME FOR STRUCTURAL PURPOSES[1]

Serial Designation: C 5 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1921; REVISED, 1922, 1924

I. REQUIREMENTS

Requirements. 1. The quicklime shall conform to the following requirements as to chemical composition, calculated to the non-volatile basis:

	CALCIUM LIME	MAGNESIUM LIME
Calcium oxide, minimum, per cent	75	..
Magnesium oxide, minimum, per cent	..	20
Calcium and magnesium oxides, minimum, per cent	95	95
Silica, alumina, and oxide of iron, maximum, per cent	5	5
Carbon dioxide, maximum, per cent		
(*a*) If sample is taken at the kiln	3	3
(*b*) If sample is taken at any other place	10	10

Percentage of Residue. 2. When tested in accordance with the method described in section 4, quicklime shall contain not more than 15 per cent by weight of residue.

II. METHODS OF TEST

Chemical Analysis. 3. The chemical analysis of the lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 – 25 T) of the American Society for Testing Materials.[2]

Percentage of Residue. 4. An average $2\frac{1}{2}$-kg. (5-lb.) sample shall be put in a box of wood or of some material of similarly low thermal conductivity and slaked by an experienced operator with sufficient water at 70 to 80° F.

[1] These Tentative Specifications are in effect a revision of the Standard Specifications for Quicklime. The Standard Specifications, which were last published under the Serial Designation: C 5–15, have accordingly been withdrawn.

Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

[2] See p. 233.

(21 to 27° C.) to produce the maximum quantity of lime putty, care being taken to avoid "burning" or "drowning" the lime. It shall be allowed to stand for 1 hour and then washed through a No. 20 sieve by a stream of water having a moderate pressure. No material shall be rubbed through the sieve. The washing shall be continued until the residue on the screen appears to consist wholly of coarse particles, but in no case shall washing be continued more than 30 minutes. The residue shall be dried to constant weight at a temperature of 212 to 225° F. (100 to 107° C.). The sample of lump lime taken for this test shall be broken so as to all pass a 1-in. ring and be retained on a $\frac{1}{4}$-in. sieve. Pulverized lime shall be tested as received.

III. SAMPLING, RETESTING, PACKING AND MARKING

Sampling, Retesting, Packing, Marking.

5. The sampling, retesting, packing and marking shall be conducted in accordance with the Tentative Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50 – 24 T) of the American Society for Testing Materials.[1]

APPENDIX

Quicklime can never be used as such for structural purposes; it must always be slaked first. Since the method of slaking is an important factor in determining the quality of the finished product, the following directions are given, not as a part of the specifications, but as information for the further protection of the purchaser.

PREPARATION OF LIME PUTTY

Directions for Slaking.

1. (*a*) *Introduction.*—Different kinds of lime vary considerably in the way in which they behave with water. A little supervision over the operation of slaking will amply pay for itself by insuring the production of the greatest possible quantity and the best possible quality of putty. To find out how to slake a new lot of lime, it is safest to try a little of it and see how it works. Since different lots of the same brand of lime vary somewhat, and since the weather conditions at the time have a decided influence, it is wise to try a sample from each lot used, whether familiar with the brand or not.

(*b*) *Classification of Limes.*—In a bucket, put two or three lumps of lime about the size of one's fist, or, in the case of granular lime, an equivalent amount. Add enough water to just barely cover the lime, and note how long it takes for slaking to begin. Slaking has begun when pieces split off from the lumps or when the lumps crumble. Water of the same temperature should be used for test and field practice.

If slaking begins in less than five minutes, the lime is quick slaking; from five to thirty minutes, medium slaking; over thirty minutes, slow slaking.

[1] See p. 246.

(*c*) *Directions for Slaking.*—For quick-slaking lime, always add the lime to the water, not the water to the lime. Have enough water at first to cover all the lime completely. Have a plentiful supply of water available for immediate use—a hose throwing a good stream, if possible. Watch the lime constantly. At the slightest appearance of escaping steam, hoe thoroughly and quickly, and add enough water to stop the steaming. Do not be afraid of using too much water with this kind of lime.

For medium-slaking lime, add the water to the lime. Add enough water so that the lime is about half submerged. Hoe occasionally if steam starts to escape. Add a little water now and then if necessary to prevent the putty from becoming dry and crumbly. Be careful not to add any more water than required, and not too much at a time.

For slow-slaking lime, add enough water to the lime to moisten it thoroughly. Let it stand until the reaction has started. Cautiously add more water, a little at a time, taking care that the mass is not cooled by the fresh water. Do not hoe until the slaking is practically complete. If the weather is very cold, it is preferable to use hot water, but if this is not available, the mortar box may be covered in some way to keep the heat in.

Preparation of Putty for Use.

2. (*a*) *White Coat.*—After the action has ceased, run off the putty through a No. 10 sieve and store for a minimum of two weeks.

(*b*) *Base Coats.*—After the action has ceased, run off the putty through a No. 8 sieve. Add sand up to equal parts by weight, all of the hair required, and store for a minimum of two weeks.

(*c*) *Masons' Mortar.*—After the action has ceased, add part or all of the sand required, and store for a minimum of 24 hours.

TENTATIVE SPECIFICATIONS

FOR

QUICKLIME FOR USE IN THE MANUFACTURE OF SULFITE PULP[1]

Serial Designation: C 46 – 25 T

This is a Tentative Standard only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1924, 1925.

Lime in Manufacture of Sulfite Pulp.

1. Lime is used in the "Milk of Lime" or "Tank System" of sulfite pulp manufacture for making the cooking liquor. The milk of lime is held in solution or suspension in a series of tanks equipped with suitable agitators. The sulfur dioxide is forced or drawn through these tanks successively. In some cases, the tanks are built on top of each other in the form of a tower. The contents of the first tank are drawn off when the liquor has reached a certain strength (3.5 to 6 per cent total SO_2) and the contents of the second and third tanks progress to the first and second tanks respectively. The third tank is again charged with fresh milk of lime. There are other systems of absorption which provide for continuous instead of intermittent operation. The function of the lime is to furnish the base for the formation of the bisulfites of calcium and magnesium.

I. REQUIREMENTS.

Requirements.

2. (*a*) Either calcium or magnesium lime may be used, but owing to the greater solubility and reactivity of magnesium bisulfite, high magnesium lime gives the best results. The ratio of MgO to CaO shall be reasonably constant and the lime shall be moderately free from impurities.

(*b*) The quicklime shall conform to the following requirements

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

as to chemical composition, all percentages to be based on the weight of the sample taken at the point of manufacture:[1]

	Calcium Lime Maximum	Calcium Lime Minimum	Magnesium Lime Maximum	Magnesium Lime Minimum
Calcium oxide, per cent	....	92.5	....	55.4
Magnesium oxide, per cent	2.5	....	....	39.6
Oxides of silicon, iron and aluminum, per cent	3.0	....	3.0	....

II. METHODS OF TEST.

Chemical Analysis.

3. The chemical analysis of the lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 – 25 T) of the American Society for Testing Materials.[2]

III. SAMPLING, INSPECTION, REJECTION, ETC.

Sampling, Inspection, Rejection, etc.

The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Tentative Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50 – 24 T) of the American Society for Testing Materials.[3]

[1] If it is desired to use the non-volatile basis the figures given above should be changed to the following:

	Calcium Lime Maximum	Calcium Lime Minimum	Magnesium Lime Maximum	Magnesium Lime Minimum
Calcium oxide, per cent	....	94.3	....	56.5
Magnesium oxide, per cent	2.6	....	....	40.4
Oxides of silicon, iron and aluminum, per cent	3.1	....	3.1	....

[2] See p. 233.

[3] See p. 246.

TENTATIVE SPECIFICATIONS
FOR
HYDRATED LIME
FOR THE MANUFACTURE OF VARNISH.[1]

Serial Designation: C 47 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922.

Lime in the Manufacture of Varnish.

1. Hydrated lime is used in the manufacture of oleo-resinous varnishes to harden and partially neutralize the rosin. About 8 lb. of hydrated lime are gradually added to 100 lb. of the melted rosin, and heated for a short period.

I. REQUIREMENTS.

Requirements.

2. (*a*) The hydrated lime shall be of such a color that no pronounced darkening of the finished varnish will result. It shall be of such a texture and of such a fineness that all portions of it will rapidly come into intimate contact with the resins so as to enter into the chemical reactions with rapidity.

(*b*) The lime shall be completely hydrated[2] since a high degree of hydration is important.

(*c*) *Fineness.*—Not more than 10 per cent of the hydrated lime shall be retained on a No. 230 sieve.

(*d*) *Reactivity.*—The hydrated lime shall conform to the requirements of the tung-oil heat test as indicated in Section 5.

(*e*) The hydrated lime shall conform to the following requirements as to chemical composition, calculated to the non-volatile basis:

Calcium oxide (CaO), minimum	94.0	per cent
Magnesium oxide (MgO), maximum	3.0	"
Iron oxide, alumina oxide and insoluble matter, maximum	3.0	"
Iron oxide (Fe_2O_3), maximum	0.4	"
Carbon dioxide (CO_2), maximum:		
(*a*) If sample is taken at place of manufacture	3.0	"
(*b*) If sample is taken at other than place of manufracture	5.0	"

II. METHODS OF TEST.

Chemical Analysis.

3. The chemical analysis of the lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone, Quick-

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

[2] A very high calcium lime is preferred. It is especially important that the iron content be low in order to avoid undesirable color effects.

lime and Hydrated Lime (Serial Designation: C 25 – 25 T) of the American Society for Testing Materials.[1]

Fineness. 4. One hundred grams of the sample as received shall be placed on a No. 230 sieve.[2] The material shall be washed by means of a stream of water from a faucet (Note 1). Washing shall be continued until the water coming through the sieve is clear. The residue upon the No. 230 sieve shall be dried to constant weight in an atmosphere free from CO_2 in a drying oven whose temperature is maintained between 100 and 120° C. (212 and 248° F.). The weight of this residue shall be calculated as percentage of the original sample.

NOTE 1.—A small piece of rubber tubing attached to a water faucet will be found convenient. The velocity of the stream of water may be increased by pinching the tube, but the velocity should not be sufficient to cause any danger of splashing the sample over the sides of the sieve.

Tung Oil Heat Test. 5. Fifty grams of raw tung oil[3] shall be placed in a 100-cc. beaker and 1 g. of hydrated lime stirred in. A cloudy mass will result. Heat shall then be applied. High-quality hydrated lime will usually start to react at approximately 80° C. (176° F.), small bubbles rising to the surface. As heating is continued, large flakes of lime tungate will form and rise to the surface to form a thick white scum and the oil below will gradually become fairly clear. These changes will usually be completed when a temperature of 120° C. (248° F.) is reached. The heating shall be continued until the mass reaches a temperature of 140° C. (284° F.). It shall then be allowed to cool and the formation of the thick scum on the surface noted which will, when freed of air, gradually drop to the bottom of the beaker. This scum consists of tung oil soaps of lime which are quite insoluble in the oil. Addition of rosin will readily effect solution of these soaps.

A poor grade of lime will fail to react, the absence of bubbles or flakes of lime tungate being noticeable. The oil will remain quiescent and cloudy. Only small amounts of soaps will form on the surface.

III. SAMPLING, INSPECTION, REJECTION, ETC.

Sampling, Inspection, Rejection, etc. 6. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Tentative Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50 – 24 T) of the American Society for Testing Materials.[4]

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[1] See p. 233.

[2] For detailed specifications for these sieves, see U. S. Bureau of Standards *Letter Circular No. 74*.

[3] Tung oil for this test should conform to the requirements of the Tentative Specifications for Raw Tung Oil (Serial Designation: D 12 – 25 T) of the American Society for Testing Materials. See p. 266.

[4] See p. 246.

TENTATIVE SPECIFICATIONS
FOR
QUICKLIME FOR USE IN WATER TREATMENT[1]

Serial Designation: C 53 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

1. (*a*) In the treatment of water for public supplies, lime is used alone or with iron sulfate or aluminum sulfate to produce a precipitate which assists in the clarification of the water and in the removal of the bacteria by filtration. An excess of lime is sometimes used to remove part of the hardness of the water. **Quicklime for Water Treatment.**

(*b*) Lime and soda ash are used together for softening water.

(*c*) The lime serves as a chemical reagent in water treatment. The only useful constituent of the lime is the calcium oxide capable of reacting with the other chemicals which are in the water or which are added to it. Inert material, besides reducing the value in proportion to its amount, also makes more sludge to be disposed of for a given amount of chemical action and thus reduces the capacity of the equipment in which it is used.

I. REQUIREMENTS

2. (*a*) The lime shall be substantially free of core, ash, and dirt, and shall be capable of disintegrating, in water, into a suspension of finely divided material. **Requirements.**

(*b*) The standard quicklime for use in water treatment shall contain 90 per cent of available lime. All percentages enumerated herein are based on the sample taken at the point of manufacture.

II. METHODS OF TEST

3. The chemical analysis for available lime shall be made in accordance with the Tentative Methods of Chemical Analysis of **Chemical Analysis.**

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

Limestone, Quicklime and Hydrated Lime (Serial Designation: C 25 – 25 T) of the American Society for Testing Materials.[1]

III. SAMPLING, INSPECTION, REJECTION, ETC.

Sampling, Inspection Rejection, etc.

4. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Tentative Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation: C 50 – 24 T) of the American Society for Testing Materials.[2]

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[1] See p. 233.
[2] See p. 246.

TENTATIVE SPECIFICATIONS

FOR

HYDRATED LIME FOR USE IN WATER TREATMENT[1]

Serial Designation: C 54 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924; Revised, 1925.

Hydrated Lime for Water Treatment.

1\. (*a*) In the treatment of water for public supplies, lime is used alone or with iron sulfate or aluminum sulfate to produce a precipitate which assists in the clarification of the water and in the removal of bacteria by filtration. An excess of lime is sometimes used to remove part of the hardness of the water.

(*b*) Lime and soda ash are used for softening water.

(*c*) Hydrated lime serves as a chemical reagent in water treatment. The only useful constituent of the hydrated lime is the calcium hydroxide capable of reacting with the other chemicals which are in the water or which are added to it. Inert material, besides reducing the value in proportion to its amount, also makes more sludge to be disposed of for a given amount of chemical action and thus reduces the capacity of the equipment in which it is used.

I. REQUIREMENTS

Requirements.

2\. The standard hydrated lime for use in water treatment shall contain 90 per cent of available calcium hydroxide (equivalent to 68.1 per cent of calcium oxide). All percentages enumerated herein are based on the sample taken at the point of manufacture.

II. METHODS OF TEST

Chemical Analysis.

3\. The chemical test for available lime shall be made in accordance with the Tentative Methods of Chemical Analysis of Limestone,

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

Quicklime and Hydrated Lime (Serial Designation: C 25 – 25 T) of the American Society for Testing Materials.[1]

III. SAMPLING, INSPECTION, REJECTION, ETC.

Sampling, Inspection, Rejection, etc.

4. The sampling, inspection, rejection, retesting, packing and marking shall be conducted in accordance with the Tentative Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (Serial Designation C 50 – 24 T) of the American Society for Testing Materials.[2]

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[1] See p. 233.
[2] See p. 246.

TENTATIVE SPECIFICATIONS

FOR

CONCRETE AGGREGATES[1]

Serial Designation: C 33 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

Issued, 1921; Revised, 1923.

FINE AGGREGATE

Fine Aggregate. 1. Fine aggregate shall consist of sand, stone screenings, or other inert materials with similar characteristics, or a combination thereof, having clean, hard, strong, durable, uncoated grains, free from injurious amounts of dust, lumps, soft or flaky particles, shale, alkali, organic matter, loam or other deleterious substances.

Grading. 2. Fine aggregate shall preferably be graded from fine to coarse, with the coarser particles predominating, within the following limits:

Passing $\frac{3}{8}$-in. sieve		100	per cent
Passing No. 4 sieve		85	"
Passin No. 50 sieve	not more than	30	"
Weight removed by decantation test	not more than	3	"

The sieves shall conform to the requirements specified in the Standard Method of Test for Sieve Analysis of Aggregates for Concrete (Serial Designation: C 41) of the American Society for Testing Materials.[2]

Strength in Concrete. 3. The fine aggregate shall be tested in combination with the coarse aggregate and the cement with which it is to be used and in the proportions, including water, in which they are to be used on the work, in accordance with the requirements specified in Section 6. In case the test provided in Section 6 shows the strengths specified therein, the fine aggregate shall be considered acceptable.

Mortar Strength. 4. With the approval and consent of the engineer, the following requirements may be substituted:

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. J. C. Pearson, Secretary of Committee C-9 on Concrete and Concrete Aggregates, Lehigh Portland Cement Co., Allentown, Pa.

[2] 1924 Book of A.S.T.M. Standards.

(*a*) Mortar briquettes, cylinders or prisms, consisting of one part by weight of portland cement and three parts by weight[1] of fine aggregate, mixed and tested in accordance with the methods described in the Standard Specifications and Tests for Portland Cement (Serial Designation: C 9) of the American Society for Testing Materials[2] for tension tests and in accordance with the Tentative Specifications and Tests for Compressive Strength of Portland-Cement Mortars (Serial Designation: C 9 – 16 T) of the American Society for Testing Materials[3] for compression tests shall show a tensile or compressive strength at the age of 7 and 28 days not less than that of 1:3 standard Ottawa sand mortar of the same consistency made with the same cement.

NOTE.—In testing aggregates, care should be exercised to avoid the removal of any coating on the grains which may affect the strength; bank sand should not be dried before being made into mortar, but should contain natural moisture. The percentage of moisture may be determined upon a separate sample and the weight of the sand used in the test corrected for the moisture content.

(*b*) Upon failure to meet this requirement, the proportion of cement in the concrete mixture shall be increased or the proportions of cement, fine aggregate, coarse aggregate and water changed in such a way as to produce the strength specified in Section 6.

Colorimetric Test.

5. No fine aggregate showing a color darker than the standard color when tested in accordance with the Standard Method of Test for Organic Impurities in Sands for Concrete (Serial Designation: C 40) of the American Society for Testing Materials[2] shall be used unless the strength requirement of Sections 4 or 6 is fulfilled.

Concrete Tests.

6. The grade of concrete required with its compressive strength in pounds per square inch shall be specified by the engineer. The concrete materials, including cement, fine aggregate, coarse aggregate and water, mixed in the proportions in which they are to be used in the work, and tested in accordance with the standard methods of test, shall at 28 days develop a strength of not less than that specified for the grade of concrete required. Upon failure to meet this requirement, the proportion of cement in the concrete mix shall be increased or the proportions of cement, fine aggregate, coarse aggregate and water shall be changed in such a way as to produce the specified strength.

COARSE AGGREGATE

Coarse Aggregate.

7. Coarse aggregate shall consist of crushed stone, gravel, air-cooled blast-furnace slag, or other approved inert materials with

[1] When approved by the engineer, these proportions may be by volume, assuming 1 cu. ft. of cement to weigh 94 lb.

[2] 1924 Book of A.S.T.M. Standards.

[3] See p. 193.

similar characteristics, or a combination thereof, having clean, hard, strong, durable, uncoated pieces free from injurious amounts of soft, friable, thin, elongated or laminated pieces, alkali, organic or other deleterious matter.

8. In the case of blast-furnace slag, the weight per cubic foot shall not be less than 65 lb. per cu. ft. for use in concrete structures not subject to abrasion, and not less than 70 lb. per cu. ft. for use in concrete floor or road construction. **Quality.**

9. (*a*) Coarse aggregate shall be well graded from fine to coarse within the following limits:[1] **Grading of Coarse Aggregate.**

Passing .... in. sieve (maximum size).............. 95 per cent
Passing .... in. sieve (intermediate size)........ .... to .... per cent

Not more than 15 per cent shall pass the No. 4 sieve; not more than 5 per cent shall pass the No. 8 sieve.

(*b*) The maximum size of coarse aggregate shall be either designated by the engineer or indicated on the plans.

NOTE.—The grading as above specified is intended to secure uniformity of aggregate, but will be governed by local conditions. The following table indicates desirable gradings, in percentes, for coarse aggregate for certain maximum sizes:

Maximum Size of Aggregate, in.	Square Openings, in.							Per Cent Passing No. 4 Sieve, not more than	Per Cent Passing No. 8 Sieve, not more than
	3	2½	2	1½	1	¾	½		
3	95	..	..	40–75	..	..	..	15	5
2½	..	95	..	..	..	..	..	15	5
2	..	..	95	..	40–75	..	..	15	5
1½	..	..	..	95	..	40–75	..	15	5
1	..	..	..	..	95	..	40–75	15	5
¾	..	..	..	..	..	95	..	15	5

[1] When there are several suitable aggregates available, a thorough investigation of the relative economy of each for producing concrete of the desired strength is advisable, especially for work of considerable magnitude.

TENTATIVE SPECIFICATIONS
FOR
CONCRETE BUILDING BRICK[1]
Serial Designation: C 55 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924.

I. DEFINITION

Definition. 1. *Concrete Brick.*—For the purpose of these specifications, brick made of portland-cement concrete.

II. STANDARD SIZE

Standard Size. 2. The standard size of concrete building brick shall be $2\frac{1}{4}$ by $3\frac{3}{4}$ by 8 in.

III. SAMPLING

Sampling. 3. For the purpose of test, bricks shall be selected by an experienced person so as to represent the commercial product. All bricks shall be carefully examined and their condition noted before being subjected to any kind of test. For the purpose of the tests, fifteen bricks will be required; they shall be thoroughly dried to constant weight in a suitable oven at a temperature of from 225 to 250° F. (107 to 121° C.).

IV. PHYSICAL TESTS

Absorption. 4. (*a*) At least five dry bricks shall be weighed and completely submerged in water at a temperature between 60 and 80° F. (15 and 27° C.). The water shall be heated to boiling within one hour, boiled continuously for five hours and then allowed to cool to a temperature between 60 and 80° F. (15 and 27° C.). The bricks shall then be removed, the surface water wiped off with a damp cloth and the bricks quickly weighed.

(*b*) The percentage of absorption shall be calculated on the dry weight from the formula:

$$\text{Percentage of Absorption} = \frac{100\,(B-A)}{A} \times \frac{C}{125}$$

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. W. E. Emley, Secretary of Committee C-3 on Brick, U. S. Bureau of Standards, Washington, D. C.

where A = the weight of the dry brick, B = the weight of the saturated brick, and C = the weight of the dry brick in pounds per cubic foot.

NOTE.—The factor $\frac{C}{125}$ is introducted to correct for variations in the weight per cubic foot of the concrete of which the brick is made.

5. (*a*) Compression tests shall be made on at least five whole bricks, previously dried. The specimens shall be tested flat. To secure a uniform bearing in the testing machine, the flat surfaces shall be bedded in a thin coat of calcined gypsum (plaster of Paris), **Compression Tests.**

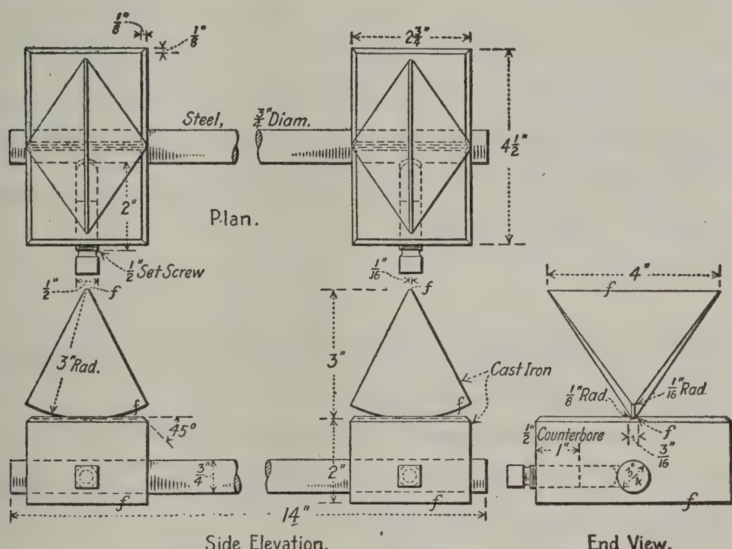

FIG. 1.

or in a mixture of calcined gypsum and portland cement, spread upon plate glass previously coated with a thin film of oil. Before applying the plaster, the bearing surfaces of the brick shall receive a coating of shellac. The brick shall be pressed firmly upon the surface, making the layer as thin as possible, and shall be permitted to remain undisturbed until set. The depressions of recessed or paneled bricks shall be filled with neat portland-cement mortar, which shall stand at least 24 hours before testing.

(*b*) The machine used for the compression tests shall be equipped with a spherical bearing block kept thoroughly lubricated to insure accurate adjustment, which should be made by hand under a small

initial load. The spherical bearing block shall be used on top of the specimen. During the test, the beam of the testing machine shall be kept constantly in a floating position.

(*c*) The breaking load shall be divided by the area in compression and the results reported in pounds per square inch.

Transverse Tests.

6. (*a*) At least five bricks, previously dried, shall be tested, laid flat-wise on knife edges with a span of 7 in. and with the load applied at midspan. Steel bearing plates, $\frac{1}{4}$ in. in thickness by $1\frac{1}{2}$ in. in width, shall be placed between the knife edges and the brick. The upper edges of the knife edges shall be straight and have full contact with the $\frac{1}{4}$-in. bearing plates. They shall have a length at least equal to the width of the brick to be tested. Their bases shall be so curved or rounded as to permit free movement of the upper edges both longitudinally and transversely. The use of knife edges of the type shown in Fig. 1 is recommended, although other types may be used which will provide free movement at the ends and at the same time insure full and even bearing at all times during the test.

(*b*) The modulus of rupture shall be calculated in pounds per square inch from the formula:

$$R = \frac{3\,Wl}{2\,bd^2}$$

in which l = the distance between the supports in inches, b = the breadth and d = the depth of the brick in inches, and W = the load in pounds at which the brick failed.

Records of Test Results.

7. In recording the results of the tests the type of brick shall be defined, whether dry pressed, or wet mixed. It is recommended that the data obtained be recorded as indicated on the accompanying "Laboratory Record."

V. TEST REQUIREMENTS

Test Requirements.

8. (*a*) When sampled at the plant, the age of concrete bricks when tested shall be not less than 28 days nor more than 60 days. Tests may be made on the brick delivered on the job.

(*b*) The bricks shall meet the following physical requirements:

Kind of Brick	Absorption Limits, per cent		Compressive Strength (flat), lb. per sq. in.		Modulus of Rupture, lb. per sq. in.	
	Mean of 5 Tests, Maximum	Individual Maximum	Mean of 5 Tests, Minimum	Individual Minimum	Mean of 5 Tests, Minimum	Individual Minimum
Face brick..................	10	12	1500	1000	300	250
Common brick.............	12	15	1500	1000	300	250

LABORATORY RECORD

THE ENGINEERING LABORATORY OF..

Brick received from..

Address..

Sampled by.................................... Date....................................

Type.................................... Class (A.S.T.M.)....................................

Appearance of bricks..

ABSORPTION TEST

Sample No.	Weight			Absorption, per cent $\frac{100\ B\text{-}A}{A} \times \frac{C}{125}$	Remarks
	Dry (A)	Saturated (B)	Difference $(B\text{-}A)$		
1					
2					
3					
4					
5					
Sum					
Average					

COMPRESSION TEST

(WHOLE BRICKS TESTED FLAT.)

Sample No.	Dimensions of Brick			Load		Compressive Strength, $\left(\frac{L}{a}\right)$ lb. per sq. in.	Remarks
	Width (b), in.	Length (l), in.	Area (a) $a=bl$	At First Crack, lb.	At Failure (L), lb.		
1							
2							
3							
4							
5							
Sum	....	....	....	....	....		
Average	....	....	....	....	....		

LABORATORY RECORD.—*Continued.*

TRANSVERSE TEST

Sample No.	Dimensions			Load (W), lb.	Modulus of Rupture, $R=\frac{3Wl}{2bd^2}$	Remarks
	Width (b), in.	Depth (d), in.	Span (l), in.			
1						
2						
3						
4						
5						
Sum		....	....	....		
Average		....	....	....		

SUMMARY

Average Absorption, per cent	Average Compressive Strength, lb. per sq. in.	Average Modulus of Rupture

Observers:

------------------------------------ ------------------------------------

------------------------------------ *In charge of tests.*

TENTATIVE SPECIFICATIONS AND TESTS

FOR

HOLLOW BURNED-CLAY LOAD-BEARING WALL TILE[1]

Serial Designation: C 34 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1921; REVISED, 1924.

SPECIFICATIONS

1. These specifications apply to hollow load-bearing wall tile made from surface clay, shale, fire clay or admixtures thereof. **Application.**

I. CLASSIFICATION

2. (*a*) According to the results of physical tests, tile shall be classified as Hard, Medium, and Soft on the basis of the following requirements:[2] **Classification.**

Class	Absorption, per cent		Compressive Strength, Based on Gross Area, lb. per sq. in.[a]			
			End Construction		Side Construction	
	Mean of 5 Tests	Individual Maximum	Mean of 5 Tests	Individual Minimum	Mean of 5 Tests	Individual Minimum
Hard............	12 or less	15	1800 or more	1400	1000 or more	700
Medium.........	12 to 16	19	1800 to 1400	1000	1000 to 700	500
Soft.............	16 to 25	28	1400 to 1200	700	700 to 500	350

[a] Gross area shall be taken as the total area enclosed by the outside dimensions of the unit taken in a direction perpendicular to that in which the load is carried.

(*b*) Where end-construction tile are used on the side they shall meet the requirements of that construction, and *vice versa*.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. F. J. Huse, Secretary of Committee C-10 on Hollow Masonry Building Units, The Hollow Building Tile Association, 1409 Conway Building, Chicago, Ill.

[2] As different types of clay are used in the manufacture of tile, color cannot be taken as indicative of classification.

II. WEIGHTS

Weights.

3. (*a*) The tile shall have the following dry weights determined as hereinafter specified:

Size of Unit, in.	Number of Cells	Standard Weight, lb.
3¾ by 12 by 12	3	20
6 by 12 by 12	6	30
8 by 12 by 12	6	36
10 by 12 by 12	6	42
12 by 12 by 12	6	48
3¾ by 5 by 12	1	9
8 by 5 by 12	2	16
8 by 5 by 12 ("L" Shaped)	..	16
8 by 6¼ by 12 ("T" Shaped)	4	16
8 by 7¾ by 12 (Square)	6	24
8 by 10¼ by 12 ("H" Shaped)	7	32

(*b*) A tolerance of 5 per cent will be allowed on the above standard weights.

III. DIMENSIONS

Dimensions.

4. No dimension shall vary more than 3 per cent from the standard dimensions for any form of tile.

IV. WEATHERING RESISTANCE

Weathering Resistance.

5. All tile used in exterior work subjected to weathering conditions shall be able to withstand 100 alternate freezings and thawings. Tile classed as hard or medium by these specifications may be considered as meeting the weathering requirement, provided they are burned to the normal maturity for the given clay. Tile classed as soft shall be accepted as meeting the weathering requirement only on the basis of freezing tests.

V. FIRE RESISTANCE

Fire Resistance.

6. The tile shall meet the requirements of the Standard Specifications for Fire Tests of Materials and Construction (Serial Designation: C 19) of the American Society for Testing Materials[1] as they apply to load-bearing walls and partitions, and to be acceptable shall develop the safe resistance period specified in the contract and in all cases shall give a resistance of not less than that equivalent to the requirements for the one-hour classification where the fire resistance of the construction is limited by the properties of the tile. The manufacturer shall supply such available information on the fire test performance

[1] 1924 Book of A.S.T.M. Standards.

of the given or closely similar product as will aid the purchaser in deciding whether his requirements are met. Further tests in accordance with the above-named standards may be conducted by the purchaser.

VI. WORKMANSHIP AND FINISH

7. All tile shall be well burned, reasonably free from laminations and from such cracks, blisters, surface roughness, and other defects as would interfere with the proper setting of the tile or impair the strength or permanence of the construction. **Workmanship.**

8. The exterior surface of all tile intended for plaster or stucco shall be scored in such a manner as to give good anchorage. **Scoring.**

VII. MARKING

9. All tile shall bear the word "Load-Bearing" and initials, name or trademark of the manufacturer. These marks shall be indented on the exterior of the tile and shall be plainly legible. **Marking.**

VIII. INSPECTION AND REJECTION

10. Proper facilities shall be provided the purchaser for sampling and inspection either at the factory or at the site of the work, as may be specified in the contract. At least 10 days from the time of sampling shall be allowed for the completion of the test. The inspection shall be based on the requirements specified above. All tests shall be made in accordance with the methods hereinafter prescribed. **Inspection.**

11. (*a*) Individual tile shall be rejected for failure to meet the weight, size, workmanship and finish requirements. In case of failure to meet the absorption and strength requirements for the class specified, the seller may sort the shipment and new samples shall be selected from the retained lot and tested at his expense. In case the second set of samples fails to meet the test requirements the entire lot shall be rejected. **Rejection.**

(*b*) By agreement, acceptance may be based on dry weight of the units, percentage of absorption, fire resistance and the workmanship and finish, except that where loads approximating working-load values will be imposed on the tile construction, the tests for compressive strength shall also be made and the tile shall meet the compressive strength requirements for the given class of tile. **Acceptance.**

12. Except as specified in Section 11 and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser. **Expense of Tests.**

TESTS

IX. SAMPLING

Selection of Samples for Test. 13. Samples of tile for testing shall be selected by the purchaser or by a competent representative authorized by him to do this work. In all cases the samples shall be representative of the whole lot of tile from which they are selected. Full-size tile shall be taken in all cases.

Number of Samples. 14. For the strength, absorption, and weight determinations, five individual tile shall be selected from each kiln or from each 100-ton lot. In no case shall less than five tile be taken. For the fire test the size of the test panel will govern the number of tile required. Samples for the freezing tests shall be taken from tile that have not been subjected to strength or fire tests.

Time and Place of Selecting Samples. 15. Samples for the strength and absorption tests shall be selected at the factory or at the site of the work, as specified. If the fire or freezing tests are to be made it shall be so specified at the time of placing the order, samples for fire tests being selected at the factory at least 45 days in advance of the time of filling the order and 90 days in advance for freezing tests.

Marking Samples. 16. All tile selected for test shall be plainly and permanently marked for reference by the testing operator.

X. WEIGHT DETERMINATIONS

Weight Determinations. 17. The five tile, if not in kiln-dry condition, shall be dried to constant weight at a temperature of not less than 100° C. (212° F.) and be weighed separately. The scale shall be sensitive to within 0.5 per cent of the weight of the smallest unit.

XI. STRENGTH TESTS

Samples. 18. Five full-size dry tile shall be used.

Speed of Testing Machine. 19. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute.

Bearing Block. 20. A spherical bearing block of proper design shall be placed on top of the test sample.

Capping. 21. (*a*) Bearing surfaces of the test samples and portions adjoining them which are liable to absorb water from the plastic capping shall be coated with one coat of shellac and allowed to dry. A quantity of plastic mortar made of a mixture of three parts (by volume) of portland cement and one part of unretarded gypsum (plaster of Paris) mixed with sufficient water to spread evenly shall be placed on a plain surface which has been coated with oil, and allowed to harden suffi-

ciently to bear the weight of the tile. The surface to be capped shall be placed on this mortar, and while holding the specimen so that its axis is at right angles to the capping surface it shall be given a single firm pressure.

(*b*) The average thickness of the cap after the extruded plaster has been removed and the edges trued shall not be more than $\frac{1}{8}$ in. Patching of caps after setting shall not be permitted. Imperfect caps shall be removed and replaced with new ones.

(*c*) Where time is not available for aging the cement-gypsum cap, a cap of neat gypsum may be used, although the resulting tile strength will generally be lower than with the cement-gypsum cap. Any other method of capping which produces equally satisfactory results may be used.

Time of Testing. 22. When the cement-gypsum cap is used it shall age at least three days before the tile is tested. Where the neat gypsum cap is used the tile may be tested as soon as the plaster has been well set, but not sooner than one hour after the sample has been capped.

Position of Tile. 23. All tile shall be tested in the position in which they are designed to be used.

XII. ABSORPTION TESTS

Selection of Test Samples. 24. The samples shall consist of five tile or three representative pieces from each of these five tile. If small pieces are used, two shall be taken from the shell and one from an interior web, the weight of each piece to be not less than 227 g. ($\frac{1}{2}$ lb.). The samples shall have had their rough edges or loose particles ground off and be free from cracks from the failure of the tile in compression, where taken from tile which have been subjected to strength tests.

Marking Test Samples. 25. Each piece shall be marked so that it may be identified at any time with the tile from which it was taken. Markings which do not cover more than 5 per cent of the total superficial area of the piece shall be used.

Drying the Test Samples. 26. Preparatory to the absorption tests all samples shall be dried to constant weight in a drier or oven at a temperature of not less than 100° C. (212° F.).

Accuracy of Weighings. 27. The balance used shall be sensitive to within 0.5 per cent of the weight of the smallest unit.

Saturation of Samples. 28. After obtaining the dry weight of the samples they shall be immersed in soft, distilled or rain water, raised to the boiling point and boiled continuously for one hour, and then allowed to cool in water to room temperature.

Obtaining Saturated Weights. 29. After saturation, the sample shall be removed from the water and allowed to drain for not more than one minute. The superficial water shall be removed with a damp cloth, after which they shall be weighed immediately.

Calculating and Reporting Results. 30. The test results shall be calculated as percentages of the initial dry weight, carried to the nearest first decimal place. The results shall be reported separately for each tile, with the average for the five tile.

XIII. FREEZING TESTS

Sampling. 31. Where the freezing test is to be made, five separate representative tile shall be selected.

Preparation of Samples. 32. If not possible to use the whole tile, a piece consisting of a cell not less than 4 in. long shall be sawed from the tile. These pieces shall be saturated by immersion in cold water for at least 72 hours prior to starting the freezing.

Method. 33. Any practical method of obtaining alternate freezings and thawings may be used, the freezings to be always made with fully saturated samples and the time and temperature to be such as to insure full freezing and thawing throughout the specimen. The initial weighing and all weighings for loss shall be made on dry specimens.

Interpretation of Results. 34. Failure under the freezing and thawing treatment shall be considered to be reached when:

(*a*) The samples show superficial disintegration or spalling with loss of weight of more than 5 per cent of the initial weight; or

(*b*) The samples are badly cracked; or

(*c*) The samples show evident loss of structural strength.

TENTATIVE SPECIFICATIONS AND TESTS

FOR

HOLLOW BURNED-CLAY FIREPROOFING, PARTITION AND FURRING TILE[1]

Serial Designation: C 56 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924.

SPECIFICATIONS

1. These specifications apply to hollow fireproofing, partition and furring tile made from surface clay, shale, fire clay or admixtures thereof. **Application.**

I. CLASSIFICATION

2. (*a*) According to the results of physical tests, tile shall be classified as Hard, Medium, and Soft on the basis of the following requirements:[2] **Classification.**

Class	Absorption, per cent		Compressive Strength Based on Net Area, lb. per sq. in.			
			End Construction		Side Construction	
	Mean of 5 Tests	Individual Maximum	Mean of 5 Tests	Individual Minimum	Mean of 5 Tests	Individual Minimum
Hard	12 or less	15	3000 or more	2500	2000 or more	1500
Medium	12 to 20	25	3000 to 2000	1500	2000 to 1250	1000
Soft	20 to 28	35	2000 to 1000	750	1250 to 750	500

(*b*) Where end-construction tiles are used on the side they shall meet the requirements of that construction, and *vice versa*.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. F. J. Huse, Secretary of Committee C-10 on Hollow Masonry Building Units, The Hollow Building Tile Association, 1409 Conway Building, Chicago, Ill.

[2] As different types of clay are used in the manufacture of tile, color cannot be taken as indicative of classification.

II. WEIGHTS

Weights. 3. (*a*) The tile shall have the following dry weights determined as hereinafter specified:

Standard Partition Tile

Dimensions, in.	Number of Cells	Standard Weight, lb.
3 by 12 by 12	3	15
4 by 12 by 12	3	16
6 by 12 by 12	3	22
8 by 12 by 12	4	30
10 by 12 by 12	4	36
12 by 12 by 12	4	40

(*b*) A tolerance of 5 per cent will be allowed on the above standard weights.

III. DIMENSIONS

Dimensions. 4. No dimension shall vary more than 3 per cent from the standard dimensions for any form of tile.

IV. FIRE RESISTANCE

Fire Resistance. 5. In cases where the fire resistance is an essential property, the tile shall meet the requirements of the Standard Specifications for Fire Tests of Materials and Construction (Serial Designation: C 19) of the American Society for Testing Materials,[1] as they apply for the construction in which the tile are used. The purchaser shall specify the degree of fire resistance required (fire-resistance period), and the manufacturer shall supply such available information on fire test performance of the given or closely similar product as will aid the purchaser in deciding whether the requirements are met. Further tests in accordance with the above-named standards may be conducted by the purchaser.

V. WORKMANSHIP AND FINISH

Workmanship. 6. All tile shall be well burned, reasonably free from laminations and from such cracks, blisters, surface roughness and other defects as would evidently interfere with the proper setting of the tile or impair the permanence of the construction.

Scoring. 7. The exterior surface of all tile intended for plaster shall be scored in such a manner as to give good anchorage.

[1] 1924 Book of A.S.T.M. Standards.

VI. MARKING

8. All tile shall bear the initials, name or trademark of the manufacturer. Hollow tile intended for use in partitions shall also be marked "PARTITION." These marks shall be indented on the exterior of the tile and shall be plainly legible. **Marking.**

VII. INSPECTION AND REJECTION

9. Proper facilities shall be provided the purchaser for sampling and inspection either at the factory or at the site of the work, as may be specified in the contract. At least 10 days from the time of sampling shall be allowed for the completion of the test. The inspection shall be based on the requirements specified above. All tests shall be made in accordance with the methods hereinafter prescribed. **Inspection.**

10. (*a*) Individual tile shall be rejected for failure to meet the weight, size, workmanship and finish requirements. In case of failure to meet the absorption and strength requirements for the class specified, the seller may sort the shipment and new samples shall be selected from the retained lot and tested at his expense. In case the second set of samples fails to meet the test requirements, the entire lot shall be rejected. **Rejection.**

(*b*) By agreement, acceptance may be based on dry weight of the units, percentage of absorption, fire resistance and the workmanship and finish. **Acceptance.**

11. Except as specified in Section 10, and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser. **Expense of Tests.**

TESTS

VIII. SAMPLING

12. Samples of tile for testing shall be selected by the purchaser or by a competent representative authorized by him to do this work. In all cases the samples shall be representative of the whole lot of tile from which they are selected. Full-size tile shall be taken in all cases. **Selection of Samples for Test.**

13. For the strength, absorption, and weight determinations, five individual tile shall be selected from each kiln or from each 100-ton lot. In no case shall less than five tile be taken. For the fire test the size of the test panel will govern the number of tile required. **Number of Samples.**

14. Samples for the strength and absorption tests shall be selected at the factory or at the site of the work, as specified in the contract. If the fire tests are to be made, it shall be so specified at the time of placing the order, samples being selected at the factory at least 45 days in advance of the time of filling the order. **Time and Place of Selecting Samples.**

Marking Samples.

15. All tile selected for test shall be plainly and permanently marked for reference by the testing operator.

IX. WEIGHT DETERMINATIONS

Weight Determinations.

16. The five tile, if not in kiln-dry condition, shall be dried to constant weight at a temperature of not less than 100° C. (212° F.) and be weighed separately. The scale shall be sensitive to within 0.5 per cent of the weight of the smallest unit.

X. STRENGTH TESTS

Samples.

17. Five full-size dry tile shall be used.

Speed of Testing Machine.

18. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute.

Bearing Block.

19. A spherical bearing block of proper design shall be placed on top of the test sample.

Capping.

20. (*a*) Bearing surfaces of the test samples and portions adjoining them which are liable to absorb water from the plastic capping shall be coated with one coat of shellac and allowed to dry. A quantity of plastic mortar made of a mixture of three parts (by volume) of portland cement and one part of unretarded gypsum (plaster of Paris) mixed with sufficient water to spread evenly shall be placed on a plain surface which has been coated with oil, and allowed to harden sufficiently to bear the weight of the tile. The surface to be capped shall be placed on this mortar, and while holding the specimen so that its axis is at right angles to the capping surface it shall be given a single firm pressure.

(*b*) The average thickness of the cap after the extruded plaster has been removed and the edges trued shall not be more than $\frac{1}{8}$ in. Patching of caps after setting shall not be permitted. Imperfect caps shall be removed and replaced with new ones.

(*c*) Where time is not available for aging the cement-gypsum cap, a cap of neat gypsum may be used, although the resulting tile strength will generally be lower than with the cement-gypsum cap. Any other method of capping which produces equally satisfactory results may be used.

Time of Testing.

21. When the cement-gypsum cap is used it shall age at least three days before the tile is tested. Where the neat gypsum cap is used the tile may be tested as soon as the plaster has been well set, but not sooner than one hour after the sample has been capped.

Position of Tile.

22. All tile shall be tested in the position in which they are designed to be used.

XI. ABSORPTION TESTS

23. The samples shall consist of five tile or three representative pieces from each of these five tile. If small pieces are used two shall be taken from the shell and one from an interior web, the weight of each piece to be not less than 227 g. ($\frac{1}{2}$ lb.). The samples shall have had their rough edges or loose particles ground off and be free from cracks from the failure of the tile in compression, where taken from tile which have been subjected to strength tests. **Selection of Test Samples.**

24. Each piece shall be marked so that it may be identified at any time with the tile from which it was taken. Markings which do not cover more than 5 per cent of the total superficial area of the piece shall be used. **Marking Test Samples.**

25. Preparatory to the absorption tests all samples shall be dried to constant weight in a drier or oven at a temperature of not less than 100° C. (212° F.). **Drying the Test Samples.**

26. The balance used shall be sensitive to within 0.5 per cent of the weight of the smallest unit. **Accuracy of Weighings.**

27. After obtaining the dry weight of the samples they shall be immersed in soft, distilled or rain water, raised to the boiling point and boiled continuously for one hour, and then allowed to cool in water to room temperature. **Saturation of Samples.**

28. After saturation, the sample shall be removed from the water and allowed to drain for not more than one minute. The superficial water shall be removed with a damp cloth, after which they shall be weighed immediately. **Obtaining Saturated Weights.**

29. The test results shall be calculated as percentages of the initial dry weight, carried to the nearest first decimal place. The results shall be reported separately for each tile, with the average for the five tile. **Calculating and Reporting Results.**

TENTATIVE SPECIFICATIONS AND TESTS

FOR

HOLLOW BURNED-CLAY FLOOR TILE[1]

Serial Designation: C 57 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

SPECIFICATIONS

Application. 1. These specifications apply to hollow floor tile made from surface clay, shale, fire clay or admixtures thereof.

I. CLASSIFICATION

Classification 2. (*a*) According to the results of physical tests, tile shall be classified as Hard, Medium, and Soft on the basis of the following requirements:[2]

Class	Absorption, per cent		Compressive Strength Based on Net Area, lb. per sq. in.			
			End Construction		Side Construction	
	Mean of 5 Tests	Individual Maximum	Mean of 5 Tests	Individual Minimum	Mean of 5 Tests	Individual Minimum
Hard	12 or less	15	4000 or more	3200	2500 or more	2000
Medium	12 to 16	19	4000 to 3000	2400	2500 to 1800	1400
Soft	16 to 25	28	3000 to 2200	1700	1800 to 1300	1000

(*b*) Where end-construction tile are used on the side they shall meet the requirements of that construction, and *vice versa*.

II. WEIGHTS

Weights. 3. (*a*) The tile shall have the following dry weights determined as hereinafter specified:

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. F. J. Huse, Secretary of Committee C-10 on Hollow Masonry Building Units, The Hollow Building Tile Association, 1409 Conway Building, Chicago, Ill.

[2] As different types of clay are used in the manufacture of tile, color cannot be taken as indicative of classification.

FLAT ARCH

DEPTH OF ARCH, IN.	AVERAGE WEIGHT PER SQUARE FOOT, LB.
6	26
7	29
8	32
9	35
10	38
12	42
15	50

SEGMENTAL ARCH

DEPTH OF ARCH, IN.	AVERAGE WEIGHT PER SQUARE FOOT, LB.
6	30
8	36
10	40

TILE FOR USE IN COMBINATION HOLLOW TILE AND CONCRETE CONSTRUCTION

SIZE OF UNIT, IN.	NUMBER OF CELLS	STANDARD WEIGHT, LB.
4 by 12 by 12	3	16
6 by 12 by 12	3	22
8 by 12 by 12	4	30
10 by 12 by 12	4	36
12 by 12 by 12	4	40

(*b*) A tolerance of 5 per cent will be allowed on the above standard weights.

III. DIMENSIONS

Dimensions. 4. No dimension shall vary more than 3 per cent from the standard dimensions for any form of tile.

IV. FIRE RESISTANCE

Fire Resistance. 5. The tile shall meet the requirements of the Standard Specifications for Fire Tests of Materials and Construction (Serial Designation: C 19) of the American Society for Testing Materials,[1] as they apply to hollow floor tile, and to be acceptable shall develop the safe resistance period specified in the contract. The manufacturer shall supply such available information on the fire test performance of the given or closely similar product as will aid the purchaser in deciding whether his requirements are met. Further tests in accordance with the above named standards may be conducted by the purchaser.

[1] 1924 Book of A.S.T.M. Standards.

V. WORKMANSHIP AND FINISH

Workmanship. 6. All tile shall be well burned, reasonably free from laminations and from such cracks, blisters, surface roughness and other defects as would interfere with the proper setting of the tile or impair the strength or permanence of the construction.

Scoring. 7. The exterior surface of all tile intended for plaster shall be scored in such a manner as to give good anchorage.

VI. MARKING

Marking. 8. All tile shall bear the initials, name or trademark of the manufacturer. These marks shall be indented on the exterior of the tile and shall be plainly legible.

VII. INSPECTION AND REJECTION

Inspection. 9. Proper facilities shall be provided the purchaser for sampling and inspection either at the factory or at the site of the work, as may be specified. At least 10 days from the time of sampling shall be allowed for the completion of the test. The inspection shall be based on the requirements specified above. All tests shall be made in accordance with the methods hereinafter prescribed.

Rejection. 10. (*a*) Individual tile shall be rejected for failure to meet the weight, size, workmanship and finish requirements. In case of failure to meet the absorption and strength requirements for the class specified, the seller may sort the shipment and new samples shall be selected from the retained lot and tested at his expense. In case the second set of samples fails to meet the test requirements the entire lot shall be rejected.

Acceptance. (*b*) By agreement, acceptance may be based on dry weight of the units, percentage absorption, fire resistance and the workmanship and finish.

Expense of Tests. 11. Except as specified in Section 10 and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

TESTS

VIII. SAMPLING

Selection of Samples for Test. 12. Samples of tile for testing shall be selected by the purchaser or by a competent representative authorized by him to do this work. In all cases the samples shall be representative of the whole lot of tile from which they are selected. Full-size tile shall be taken in all cases.

13. For the strength, absorption, and weight determinations, five individual tile shall be selected from each kiln or from each 100-ton lot. Where tile of several designs are used, enough samples shall be weighed to determine the true average weight in pounds per square foot for the construction. In no case shall less than five tile be taken. For the fire test the size of the test panel will govern the number of tile required. Samples for the freezing tests shall be taken from tile that have not been subjected to strength or fire tests. **Number of Samples.**

14 Samples for the strength and absorption tests shall be selected. at the factory or at the site of the work, as specified in the contract. If the fire tests are to be made, it shall be so specified at the time of placing the order, samples being selected at the factory at least 45 days in advance of the time of filling the order. **Time and Place of Selecting Samples.**

15. All tile selected for test shall be plainly and permanently marked for reference by the testing operator. **Marking Samples.**

IX. WEIGHT DETERMINATIONS

16. The five tile, if not in kiln-dry condition, shall be dried to constant weight at a temperature of not less than 100° C. (212° F.) and be weighed separately. The scale shall be sensitive to within 0.5 per cent of the weight of the smallest unit. **Weight Determinations.**

X. STRENGTH TESTS

17. Five full-size dry tile shall be used. **Samples.**

18. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute. **Speed of Testing Machine.**

19. A spherical bearing block of proper design shall be placed on top of the test sample. **Bearing Block.**

20. (*a*) Bearing surfaces of the test samples and portions adjoining them which are liable to absorb water from the plastic capping shall be coated with one coat of shellac and allowed to dry. A quantity of plastic mortar made of a mixture of three parts (by volume) of portland cement and one part of unretarded gypsum (plaster of Paris) mixed with sufficient water to spread evenly shall be placed on a plain surface which has been coated with oil, and allowed to harden sufficiently to bear the weight of the tile. The surface to be capped shall be placed on this mortar, and while holding the specimen so that its axis is at right angles to the capping surface it shall be given a single firm pressure. **Capping.**

(*b*) The average thickness of the cap after the extruded plaster has been removed and the edges trued shall not be more than $\frac{1}{8}$ in.

Patching of caps after setting shall not be permitted. Imperfect caps shall be removed and replaced with new ones.

(c) Where time is not available for aging the cement-gypsum cap, a cap of neat gypsum may be used, although the resulting tile strength will generally be lower than with the cement-gypsum cap. Any other method of capping which produces equally satisfactory results may be used.

Time of Testing. 21. When the cement-gypsum cap is used it shall age at least three days before the tile is tested. Where the neat gypsum cap is used the tile may be tested as soon as the plaster has been well set, but not sooner than one hour after the sample has been capped.

Position of Tile. 22. In the tests, the tile shall be loaded in the direction in which they receive the principal stress in the structure.

XI. ABSORPTION TESTS

Selection of Test Samples. 23. The samples shall consist of five tile or three representative pieces from each of these five tile. If small pieces are used two shall be taken from the shell and one from an interior web, the weight of each piece to be not less than 227 g. ($\frac{1}{2}$ lb.). The samples shall have had their rough edges or loose particles ground off and be free from cracks from the failure of the tile in compression, where taken from tile which have been subjected to strength tests.

Marking Test Samples. 24. Each piece shall be marked so that it may be identified at any time with the tile from which it was taken. Markings which do not cover more than 5 per cent of the total superficial area of the piece shall be used.

Drying the Test Samples. 25. Preparatory to the absorption tests all samples shall be dried to constant weight in a drier or oven at a temperature of not less than 100° C. (212° F.).

Accuracy of Weighings. 26. The balance used shall be sensitive to within 0.5 per cent of the weight of the smallest unit.

Saturation of Samples. 27. After obtaining the dry weight of the samples they shall be immersed in soft, distilled or rain water, raised to the boiling point and boiled continuously for one hour, and then allowed to cool in water to room temperature.

Obtaining Saturated Weights. 28. After saturation, the sample shall be removed from the water and allowed to drain for not more than one minute. The superficial water shall be removed with a damp cloth, after which they shall be weighed immediately.

Calculating and Reporting Results. 29. The test results shall be calculated as percentages of the initial dry weight, carried to the nearest first decimal place. The results shall be reported separately for each tile, with the average for the five tile.

TENTATIVE METHODS OF CHEMICAL ANALYSIS OF LIMESTONE, QUICKLIME AND HYDRATED LIME.[1]

Serial Designation: C 25 – 25 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1919; REVISED, 1921, 1922, 1925.

TREATMENT OF SAMPLE

Treatment of Sample. The sample, taken and prepared in accordance with the requirements of the particular specifications of the Society applicable to the material shall be treated as follows:

Weigh out 0.5 g. of the substance and, if a limestone or hydrated lime, ignite in a covered platinum crucible in an electric muffle (Note 1) for 15 minutes, or longer if the heat is not powerful enough to effect complete decomposition within that time. Transfer to an evaporating dish, preferably of platinum (Note 2) for the sake of celerity in evaporation, mix to a thin slurry with distilled water, add 5 to 10 cc. of HCl (sp. gr. 1.20) and digest with aid of gentle heat and agitation until solution is complete. Solution may be aided and the time shortened by light pressure with the flattened end of a glass rod upon resistant lumps. Then evaporate the solution to dryness, so far as this may be possible on the water bath.

DETERMINATION OF SILICA.

Silica. When dry or nearly so, place the dish and its contents in an air bath or (covered) on a platinum triangle resting on a hot plate and, if it is a high calcium limestone that is under treatment, heat for one hour at 200° C.; if a high magnesian stone is under treatment the temperature should not exceed 120° C. Drench the cooled mass with HCl (sp. gr. 1.20) and allow to stand for a few minutes. Add an equal volume of water, cover the dish and place on the water bath for

[1] *Bulletin 700*, U. S. Geological Survey, should be used as a reference book in connection with these methods, especially when very exact and detailed analyses are desired. Blank determinations should be made on all reagents, and corrections applied when called for.

Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

10 minutes (Note 3). Separate the silica by filtration on a filter of suitable size. Wash thoroughly with dilute HCl and then wash twice with cold water.

Evaporate the filtrate to dryness. Extract the residue with HCl as before but allowing only a few minutes time. Filter this solution through a second and smaller paper.

Transfer, wet, the papers containing the separated residue to a weighed platinum crucible. Char carefully without allowing the paper to inflame and ignite to constant weight in an electric muffle and weigh (Note 1). Correct the weighed silica for foreign matter by evaporating with 5 cc. of HF and one or two drops of H_2SO_4.[1] Heat the residue for 2 or 3 minutes in an electric muffle and again weigh. The difference between this weight and that previously obtained gives the weight of the silica (Note 4).

DETERMINATION OF TOTAL IRON AND ALUMINUM.

Iron and Alumina. Fuse the residue from the silica determination using a little Na_2CO_3 (Note 5). Dissolve the cooled melt in HCl and add the solution to the filtrate from the second evaporation for silica. Add a few drops of bromine water or HNO_3 and boil the solution until all trace of bromine or chlorine is gone. Then add HCl, if not already present, sufficient to insure a total volume of 10 to 15 cc. of HCl (sp. gr. 1.20).[2] Add a few drops of methyl red solution and after diluting to 200 to 250 cc. heat the liquid to boiling. Neutralize by means of NH_4OH (dilute towards the end) until the color of the liquid changes to a distinct yellow. Boil for one or two minutes, allow to settle, filter and wash the precipitate at once two or three times with hot NH_4Cl (2-per-cent) solution and suck dry.[3]

Set the filtrate aside. Dissolve any precipitate from the paper in hot, dilute HCl, the solution passing into the beaker in which the precipitation was made and wash the paper thoroughly with hot water. Boil the solution to expel any trace of chlorine and again precipitate the hydroxides with NH_4OH as described above. Ignite the precipitate, moist (Note 1), in a weighed platinum crucible, cool and weigh as Al_2O_3 and Fe_2O_3 (Note 6).

DETERMINATION OF TOTAL IRON.

Total Iron. Fuse the combined iron and aluminum oxides (Note 7) in a platinum crucible at a very low temperature with 3 to 4 g. of $K_2S_2O_7$ or better $Na_2S_2O_7$ (Note 8). Take up the melt with so much dilute H_2SO_4 that there shall be not less than 5 g. absolute acid and enough

[1] See *Bulletin 700*, U. S. Geological Survey, p. 103.
[2] See *Bulletin 700*, U. S. Geological Survey, p. 258.
[3] See *Bulletin 700*, U. S. Geological Survey, p. 107.

water to effect solution on heating. Evaporate the solution and eventually heat until acid fumes come off copiously. After cooling and redissolving in water, filter out the small amount of silica, wash, ignite, weigh and correct by HF and H_2SO_4 (Note 9). Add the weight so corrected to the weight of silica previously found and deduct from the gross weight of the oxides of iron and aluminum.

Reduce the filtrate by zinc (Note 10). Titrate with $KMnO_4$ using a N/20 solution.

DETERMINATION OF ALUMINUM.

Subtract the calculated weight of Fe_2O_3 obtained by the method described above from the weight of $Fe_2O_3 + Al_2O_3$. Report the remainder as Al_2O_3. In case phosphorus is determined, calculate it to P_2O_5 and deduct also. **Aluminum.**

DETERMINATION OF TOTAL IRON.

ALTERNATIVE METHOD (Note 7).

Dissolve 2 to 5 g. (depending upon the amount of iron present) of the properly prepared sample in HCl and evaporate rapidly to dryness. Treat the residue with water and HCl. filter off the silica and wash several times with hot water. Precipitate the iron in a boiling solution with fixed alkali, allow to settle, filter and wash free of chlorides, using hot water. Dissolve in dilute H_2SO_4. **Total Iron, Alternative.**

Ignite the insoluble matter from the evaporated hydrochloric acid solution in a platinum crucible. Treat with H_2SO_4 and HF and heat until fumes of H_2SO_4 appear. Bring the residue into solution with the addition of a few drops more of H_2SO_4 and combine the solution with that of the bulk of the iron. Reduce this solution by zinc and titrate the iron with $KMnO_4$ in the usual manner.

DETERMINATION OF CALCIUM.

(Note 11.)

(*a*) *Gravimetric Method.*

Add a few drops of NH_4OH to the combined filtrate from the R_2O_3 precipitate, and bring the solution to boiling. To the boiling solution add 35 cc. of a saturated solution of $(NH_4)_2C_2O_4$, and continue the boiling until the precipitated CaC_2O_4 assumes a granular form; then allow it to stand 20 minutes, or until the precipitate has settled and the supernatent liquid is clear, filter and wash thoroughly with boiling water (Note 12). Place the wet filter and precipitate in a platinum crucible, and burn off the paper over the small flame of a Bunsen burner, or in a muffle. Ignite the residue, dissolve in hot dilute HCl, and make up the volume of the solution to 100 cc. with **Calcium, Gravimetric.**

water. Add NH_4OH in slight excess and boil the liquid. If a small amount of $Al_2(OH)_6$ separates, filter it out, wash with NH_4Cl (2-per-cent solution), ignite, weigh and add this weight to that found in the first determination. Then reprecipitate the lime by $(NH_4)_2C_2O_4$, allow to stand until settled, filter, wash thoroughly with boiling water (Note 12) and weigh as calcium oxide after ignition to constant weight in a covered platinum crucible.

(*b*) *Volumetric Method.* (Note 13.)

Calcium, Volumetric. Make the filtrate from the iron and alumina precipitate alkaline with NH_4OH, boil, and add 35 cc. of a boiling saturated solution of $(NH_4)_2C_2O_4$. Stir vigorously and allow to stand until the precipitate has settled, filter on an 11-cm. paper, and wash ten times with hot water (Note 14). Transfer the paper and precipitate to the beaker in which the precipitation was made, spreading the paper out against the upper portion of the beaker. Wash the precipitate from the paper with a jet of hot water, fold the paper and leave it adhering to the upper portion of the beaker. Add to the contents of the beaker 50 cc. of dilute H_2SO_4 (1:10), dilute to a volume of 250 cc. with hot water and heat to a temperature of 80 to 90° C. Titrate with the standard $KMnO_4$ solution[1] until the pink end point is obtained. Now drop the folded filter paper (which has been adhering to the side of the beaker) into the liquid; the pink color of the latter will be discharged. Finish the titration by adding $KMnO_4$, a drop at a time, until the pink end point is again obtained. A Gooch crucible may be used instead of filter paper. From the total quantity of standard $KMnO_4$ solution used, calculate the percentage of calcium oxide.

DETERMINATION OF STRONTIUM.

Strontium. Transfer the weighed oxides obtained in the gravimetric determination of calcium to a small flask of 20-cc. capacity and dissolve in HNO_3. Evaporate to dryness and heat at 150 to 160° C. Treat the thoroughly dried nitrates with as little (rarely over 2 cc.) of a mixture of equal parts of absolute alcohol and ether as may be needed to dissolve the calcium salt, solution being hastened by occasional gentle agitation. After standing over night in the corked flask collect the insoluble matter on the smallest possible filter and wash with more of the above mixture of alcohol and ether. After drying, pass a few cubic centimeters of hot water through the filter, on which may remain a few tenths of a milligram of residue, which does not usually contain any lime or other alkaline earth and whose weight is therefore to be

[1] See directions for preparing this solution.

deducted from that of the lime, unless it can be shown that it is derived from the glass of the little flask in which the nitrates of calcium and strontium were evaporated. To the solution of strontium nitrate in a small beaker add a few drops of H_2SO_4 and then its volume of alcohol, whereby the strontium is precipitated as sulfate. After twelve hours, filter on a small filter paper and wash the residue with 50-per-cent alcohol. Ignite at a low temperature, moisten with dilute H_2SO_4, evaporate, again ignite and weigh as strontium sulfate. Test the sulfate spectroscopically as to freedom from calcium and barium.

Calculate the weight of strontium oxide and deduct from the weight of calcium oxide obtained as above.

DETERMINATION OF MAGNESIUM.

Magnesium. Acidify the combined filtrates from the calcium precipitates with HCl and concentrate on the water bath to about 150 cc., and heat to boiling. To the boiling solution add 10 cc. of a saturated solution of $Na(NH_4)HPO_4$ and continue the boiling for several minutes. Then cool to room temperature, and add NH_4OH drop by drop with constant stirring until the crystallin ammonium-magnesium orthophosphate begins to form and then in moderate excess, the stirring being continued for several minutes. Allow the liquid to stand in a cool atmosphere for from 12 to 48 hours (Note 15) and filter.

Dissolve the precipitate in hot dilute HCl, make up the solution with water to about 100 cc., add 1 cc. of the saturated solution of $Na(NH_4)HPO_4$ and NH_4OH drop by drop with constant stirring until the precipitate is again formed as above described and the ammonia is present in moderate excess. Then allow it to stand in a cool atmosphere for from 12 to 48 hours (Note 15), filter on paper or a Gooch crucible, wash with dilute NH_4OH containing NH_4NO_3 (Note 16), ignite (Note 1), cool, and weigh as $Mg_2P_2O_7$ (Note 17).

DETERMINATION OF TOTAL VOLATILE MATTER.

(Loss on Ignition.)

Volatile Matter. Place 1 g. of the properly prepared sample (Note 18) in a weighed platinum crucible, cover with the lid and heat gently for 5 minutes (Note 1), gradually increasing the temperature to the maximum of the muffle, and maintain at this temperature until constant weight is obtained (usually about 15 minutes).

DETERMINATION OF MECHANICAL MOISTURE

(Limestone Only.) (Note 19.)

Mechanical Moisture. Weigh 1 g. of the properly prepared sample in a tared, wide, low-form, flat-bottomed weighing bottle. Heat uncovered in a ventilated drying oven at 120° C. for two hours. Quickly stopper

and cool in a desiccator. Lift the stopper momentarily just before weighing and weigh. The use of a similar weighing bottle as a counterpoise carried through all of the operations is a desirable procedure. The loss in weight represents "Mechanical Moisture" or "Hydroscopic Water" at 120° C.

DETERMINATION OF CARBON DIOXIDE.

Carbon Dioxide.

Carbon dioxide is to be determined upon the properly prepared sample according to the method given in U. S. Geological Survey Bulletin No. 700, p. 217. An illustration showing the arrangement of the component parts of the necessary apparatus is shown on p. 218 of that Bulletin.

With limestone, use a 0.5-g. sample; with burned lime, hydrated lime, etc., use a 5-g. sample.

Boil the weighed sample with dilute HCl in a small Erlenmeyer flask attached to an upwardly inclined condenser, whence, after passing through a drying system—calcium chloride, anhydrous copper sulfate to retain hydrogen sulfide from decomposable sulfides and any HCl that may pass over, then calcium chloride again—the carbon dioxide is caught by absorption tubes filled with soda-lime followed by calcium chloride. Of course, arrangement is made for a current of air free from CO_2 with which to sweep out the apparatus before and after the experiment, and for a slow current during its continuance.

The results are very accurate and the determination can be quickly carried out.

The manipulations are as follows: Pour hot water upon the powder in the flask fitted with a separatory funnel and delivery tube; attach this to the condenser, and force a current of air free from CO_2 through the whole system, except the weighed absorption tubes, until the original air has been displaced, the observation bulbs (containing H_2SO_4 to show the rate of gas flow) being attached directly to the drying system. Then close the stop-cock in the separatory funnel, half fill the latter with HCl (1:1), replace the rubber stopper of the funnel, insert the absorption tubes between the drying system and the observation bulbs, and allow the acid to flow into the flask, slowly if there is much carbon dioxide, rapidly if there is but little. When effervescence diminishes in the former case, at once in the latter, light the burner under the flask and start the flow of water through the condenser. Keep the flame low, so as to secure steady but quiet ebullition, and do not interrupt the air current although it should be reduced to a slow rate. With much carbon dioxide the rate of absorption is very readily noted by holding the hand to the soda-lime tubes,

which become hot or warm when absorption is taking place. Sufficient time having elapsed (Note 20), extinguish the flame, and increase the air current. When cool, disconnect the soda-lime tubes from the apparatus and allow to stand in the balance case until two weights taken 30 minutes apart agree within 0.5 mg.

The soda lime for use in this determination must be porous, not hard and unabsorptive like that sometimes used for the combustion of nitrogenous organic substances.

DETERMINATION OF SULFURIC ANHYDRIDE.

Sulfuric Anhydride. Place 2 g. of the properly prepared sample in a small, dry beaker and stir it up with 10 cc. of cold water until all lumps are broken up and the lighter particles are in suspension. Add 15 cc. of dilute HCl (1:1) and heat until solution is complete. Filter through a small paper and wash the residue thoroughly with hot water. Dilute the filtrate to 250 cc., heat to boiling, and add 10 cc. of a boiling 10-per-cent solution of barium chloride drop by drop with constant stirring. Stir well and allow to stand over night. Filter, wash with boiling water, ignite and weigh as $BaSO_4$.

DETERMINATION OF TOTAL SULFUR.

Total Sulfur. Digest 2 g. of the properly prepared sample on the hot plate with 25 to 30 cc. of bromine water. Then add 15 cc. of dilute HCl (1:1) and boil until solution is complete and all bromine has been expelled. Then proceed as outlined above under "Determination of Sulfuric Anhydride."

DETERMINATION OF PHOSPHORUS.

Phosphorus. Dissolve 10 g. of the sample in 80 cc. of dilute HNO_3 (1:1), filter, and wash the residue with hot water. Ignite, and fuse the residue with a little Na_2CO_3, cool the melt and add its nitric acid solution to the main filtrate. (Note 21.)

In case the rock is rather argillaceous, mix the powder with half its weight of Na_2CO_3 and blast strongly. Dissolve the cooled melt in HNO_3 and evaporate to dryness. Take up the dry residue with dilute HNO_3 (1:2), filter and wash the residue with hot water. Ignite the residue in platinum, and evaporate with HF and HNO_3. Finally, evaporate twice with HNO_3 alone to expel all fluorine, dissolve the residue in HNO_3 and add the solution to the main filtrate. (Note 21.)

Oxidize the nitric acid solution by boiling with 10 cc. of 1.5-per-cent $KMnO_4$ solution until manganese dioxide is precipitated. Dissolve the oxide by addition of H_2SO_3 or a sulfite free from phosphorus and boil to expel the oxides of nitrogen and sulfur. Neutralize

the HNO_3 with NH_4OH and then add 1 cc. of HNO_3 (sp. gr. 1.42) for every 100 cc. of solution. Bring the solution to a temperature of 40° C., add ammonium molybdate solution[1] and shake for 10 minutes. Allow to stand at a temperature of not over 40° C. for 1 to 12 hours. Filter and wash 10 times with a 1-per-cent KNO_3 solution. Return the precipitate to the precipitating vessel, add a measured excess of N/10 NaOH and 25 cc. of water free from CO_2 and shake or stir until the precipitate is dissolved. Dilute to 100 to 200 cc. with water free from CO_2, add 3 drops of 0.2-per-cent phenolphthalein solution, and discharge the pink color with standard acid. Finish the titration by adding standard alkali until the reappearance of the pink color. The alkali solution should be free from carbonate and be standardized against the Bureau of Standards' standard benzoic acid.[2] The ratio of phosphorus to NaOH should be considered as 1 : 23 in calculating results. (Note 22.)

DETERMINATION OF MANGANESE.

Manganese. Dissolve 10 g. of the sample in 100 cc. of dilute HNO_3 (1 : 1), filter and wash the residue with hot water. Ignite the residue in platinum, fuse with a little Na_2CO_3 and add the nitric acid solution of the melt to the main filtrate.

Dilute the solution to 150 cc. and add 0.5 g. of sodium bismuthate (Note 23). Heat for a few minutes, or until the pink color has disappeared and dioxide has precipitated. If manganese dioxide does not precipitate add more bismuthate. Clear the solution by adding a few drops of a saturated solution of sodium bisulfite or other suitable reducing agent free from chlorides, and boil to expel all oxides of nitrogen and sulphur. Cool to 15° C., add an excess of sodium bismuthate; agitate and let stand for a few minutes. Add 50 cc. of 3-per-cent HNO_3 and filter through asbestos. Wash with 3-per-cent HNO_3 until the washings run through colorless. Add a measured excess of a standard $FeSO_4$ solution and titrate back with a standard $KMnO_4$ solution of which the strength has been determined by means of the Bureau of Standards sodium oxalate. (Note 24.)

DETERMINATION OF FERROUS IRON SOLUBLE IN SULFURIC ACID.

Ferrous Iron. In limestones, the presence of carbonaceous matter renders the exact or even approximate determination of ferrous iron often impossible. Nevertheless, even in its presence acceptable results are sometimes obtainable if there is not much of such matter and if it does not give with acid a colored solution. Occasionally limestones

[1] See Blair, "The Chemical Analysis of Iron," 8th Edition, p. 92.

[2] See *Journal*, Am. Chem. Soc., Vol. 34, p. 1027 (1912), and Vol. 35, p. 1309 (1913).

show films of manganese peroxide, which likewise interferes with the determination.

(a) In Absence of Carbonaceous Matter.

The powder, one to several grams, is introduced into a stout flask of about 200 to 250-cc. capacity and boiled with a little water till all air is expelled. While still boiling, dilute H_2SO_4 is added, a little at a time, till effervescence ceases, and then a further amount. Calcium sulfate precipitates, but the iron will remain in solution. The flame is then removed and a stopper tightly inserted, through which passes a small stop-cock funnel. When cool, or nearly so, cold water is poured into the funnel, the cock cautiously opened, and the water drawn into the flask, more water being poured into the funnel as fast as it empties, till the solution amounts to 100 to 150 cc. Such precaution to exclude air is hardly necessary in most cases, however, for in presence of H_2SO_4 the oxidation of ferrous iron is exceedingly slow. It is ordinarily quite sufficient to equalize the internal and external pressures by opening the cock, removing the stopper, and pouring in cold water. The flask is then brought under a burette containing dilute $KMnO_4$ solution and the iron titrated without delay. With a carbonate which is wholly decomposable without the aid of heat, solution may be accomplished in a flask filled with CO_2, using cold acid, whereby the danger of attack of silicates is lessened if these are present. If the preference is for the $K_2Cr_2O_7$ method of titration, HCl may be used instead of H_2SO_4, in absence of manganese peroxide. In this case there is, of course, no separation of an insoluble calcium salt, a fact which renders easier the subsequent determination of the iron in any insoluble residue the rock may yield. The ferrous iron thus found is mostly, if not altogether, that existing as carbonate.[1]

(b) In Presence of Carbonaceous Matter.

Decomposition is effected in a flask by dilute H_2SO_4 in an atmosphere of CO_2. With limestones and active agitation no heat need be used, but with dolomites it will be necessary. The solution is then quickly filtered through asbestos (in an atmosphere of CO_2 if much iron is present), the residue and filter washed a few times with water, and the filtrate titrated at once with permanganate. If it is colored by organic matter, the result may be in error. As above, HCl and titration by $K_2Cr_2O_7$ may be used in absence of manganese peroxide. In either case it is important to allow the acid to act no longer than is

[1] If a determination of ferrous iron in the insoluble residue is desired see U. S. Geological Survey *Bulletin 700*, p. 265.

necessary and to filter quickly. A determination of the ferrous iron in the insoluble matter is usually not worth attempting, because of the admixed organic matter.

DETERMINATION OF AVAILABLE LIME IN QUICKLIME AND HYDRATED LIME BY THE MODIFIED SCAIFE METHOD

Available Lime. "Available lime" in quicklime or hydrated lime is that (or those) constituent (or constituents) which enters (or enter) into the reaction under the conditions of the specified method or process.

The interpretation of results obtained by the following method shall be restricted by the above definition.

Place 1.4 g. of the carefully prepared and finely ground (passing a No. 100 sieve) lime in a 400-cc. beaker, add 200 cc. of hot water, cover, heat carefully and then boil for three minutes.

Cool, wash down cover, add two drops of phenolphthalein and titrate with normal hydrochloric acid, adding the acid dropwise as rapidly as possible and stirring vigorously to avoid local excess of acid. When the pink color disappears in streaks, retard the rate of addition of acid somewhat, but continue until the pink color disappears entirely and does not reappear for 1 or 2 seconds. Note the reading and ignore the return of color.

Repeat the test, substituting for the 400-cc. beaker a 1-liter graduated flask, carrying a one-hole stopper fitted with a short glass tube drawn out to a point. Cool and add dropwise and with vigorous stirring 5 cc. less acid than before. Call the number of cubic centimeters used A. Grind up any small lumps with a glass rod flattened at one end, dilute to the mark with freshly boiled distilled water, close the flask with a solid stopper, mix thoroughly for 4 or 5 minutes and let settle for 30 minutes.

Pipette a 200-cc. portion, add phenolphthalein and titrate slowly with 0.5 N hydrochloric acid until the solution remains colorless on standing 1 minute. Call this additional number of cubic centimeters B. Then the percentage of available $CaO = 2A + 5B$.

NOTES.

To secure accurate and concordant results, it is essential that this method be followed in minute detail.

For very accurate work 0.1 N acid may be used in place of the 0.5 N acid prescribed. The calculation then becomes: Available $CaO = 2A + B$.

Standard acid solutions must be standardized by the U. S. Bureau of Standards benzoic acid No. 48-A through a sodium hydroxide solution.

PREPARATION OF STANDARD SOLUTION OF POTASSIUM PERMANGANATE.

Potassium Permanganate.

Prepare a solution of potassium permanganate containing 5.64 g. of the pure crystallized salt in each liter. Such a solution will be of such strength that 1 cc. will approximately equal 0.005 g. of CaO (Note 25).

The best and simplest way to prepare such a solution is to weigh out the required quantity of potassium permanganate and place in a bottle containing the proper volume of water a week or ten days before the solution is to be standardized. The bottle and its contents must be kept in a dark place and shaken occasionally for the first three or four days. When ready for standardization, siphon off the solution through a glass siphon into another bottle, leaving a depth of about one inch of solution undisturbed in the original bottle (Note 26). Thoroughly mix the solution in the second bottle by shaking and standardize as follows (Note 27):

Dissolve 0.5 g. of $Na_2C_2O_4$ (Note 28) in a 400-cc. beaker with 200 to 250 cc. of hot water (80 to 90° C.) and add 10 cc. of dilute H_2SO_4 (1 : 1) to the solution. Titrate at once with the permanganate solution being standardized, stirring vigorously and continuously. The permanganate solution must not be added more rapidly than 10 to 15 cc. per minute, and the last 0.5 to 1 cc. must be added dropwise, with particular care to allow each drop to be fully decolorized before the next is introduced. The excess of permanganate solution necessary to give the end point color is determined by running a blank determination in another beaker containing the same volume of acid and water present in the original beaker, and heated to the same temperature (Note 29). The value of the permanganate solution in grams of CaO per cubic centimeter of solution may be calculated from the following equations:

$$5Na_2C_2O_4 + 2KMnO_4 + 8H_2SO_4 = 5Na_2SO_4 + K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O.$$

$$5CaC_2O_4 + 2KMnO_4 + 8H_2SO_4 = 5CaSO_4 + K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O.$$

NOTES.

1. Ignitions in electric muffles are far superior to flame ignitions. If an electric muffle is not available, flame ignition and the blast lamp may be used.

2. If a platinum dish is not available, porcelain may be used. A glass container positively must *not* be used.

3. Instead of strong acid, that of half strength may be used and the heating begun at once.

4. For ordinary control work in the plant laboratory this correction may, perhaps, be neglected; the double evaporation must never be neglected.

5. Fusion with pyrosulfate is to be avoided on account of the subsequent introduction of sulfates into the solution.

6. This precipitate may also contain TiO_2, P_2O_5, and Mn_3O_4.

7. Where the iron is present in small quantities it is rather unsatisfactory to determine it in the ignited oxides from the 0.5-g. sample. Under these conditions the alternative method may be used.

8. The pyrosulfates are less troublesome and more effective than the bisulfates.

9. This correction for impurities should not be made when the hydrofluoric acid correction of the main silica determination has been omitted. After two evaporations, 1 to 2 mg. of silica are still to be found with the R_2O_3 precipitate.

10. Zinc will also reduce titanium. If this metal is to be determined and deducted from Al_2O_3, then H_2S must be used for reduction (boiling out the H_2S in a stream of CO_2 before titration). If titanium is not to be determined, the slight error introduced by its reduction with zinc is so small as to be negligible except in the most exact and detailed analyses.

11. The gravimetric method must be employed when a recovery of aluminum is desired, or when a determination of strontium is contemplated.

12. Care must be exercised in this washing, as 1000 cc. of boiling water will dissolve over 1 cg. of CaC_2O_4.

13. The volumetric method may be used for ordinary control work in the plant laboratory. It must be borne in mind that in this method all strontium oxide is reported as calcium oxide.

14. Use not more than 125 cc. for this washing.

15. The less the amount of magnesium present, the longer the precipitate must be allowed to settle.

16. Made by diluting NH_4OH with distilled water until the solution contains $2\frac{1}{2}$ per cent NH_3 by weight. Three or four drops of HNO_3 (sp. gr. 1.42) are then added.

17. When manganese is present in the limestone, it will be caught in large part with the magnesium precipitate. (See U. S. Geological Survey *Bulletin 700*, p. 134.) In case manganese was not eliminated before this precipitation, the weighed pyrophosphate must be dissolved in HNO_3, tested for manganese with bismuthate, the manganese determined and deducted as $Mn_2P_2O_7$.

18. This determination may be made if desired upon the 0.5-g. sample ignited for solution in HCl. See first page of these methods under " Treatment of Sample."

19. With burned lime and hydrated lime this determination may be omitted. Even when made in an atmosphere free from CO_2 the dried sample absorbs CO_2 sufficiently rapidly during weighing to make the result of questionable value.

20. Boiling for one minute is usually sufficient.

21. In limestones very low in phosphorus it may be deemed desirable to concentrate the phosphorus from a considerable weight of sample. In this case, proceed as directed by F. Hinden (*Zeitschr. Anal. Chemie*, Vol. 54, p. 214 (1915)), as follows: To the nitric acid solution of the large sample add a few drops of Fe_2Cl_6 solution and then NH_4OH until the liquid becomes slightly turbid. Then add 0.5 g. of pure precipitated $CaCO_3$ and boil for five minutes. Filter, dissolve the precipitate in dilute HNO_3 and proceed as above.

22. Titanium and vanadium, which interfere in this determination, should not be encountered in appreciable amounts in this class of material.

Instead of the specified alkalimetric titration method, the phosphorus in the phosphomolybdate may be determined as magnesium pyrophosphate (U. S. Geo-

logical Survey *Bulletin 700*, pp. 178–179), or by the reductor method (A. A. Blair, "Chemical Analysis of Iron," 8th Edition, pp. 91–92).

23. The persulfate method may be employed if desired.

24. The separation of manganese with the R_2O_3 precipitate by the use of bromine is not recommended on account of the incompleteness of the precipitation. Separation of manganese by means of ammonium sulfide after the ammonia precipitation is also undesirable on account of the introduction of the reagent and the slimy character of the precipitate.

See also Note 17 concerning the contamination of magnesium pyrophosphate by manganese pyrophosphate and the need of corrective measures when manganese is present in the material under analysis.

25. With high calcium limes, more than 50 cc. of this solution will probably be required to titrate the liberated oxalic acid. In such cases the use of a larger burette is to be preferred to increasing the strength of the permanganate solution.

26. The siphon should not extend nearer than 1 in. from the bottom of the original bottle.

27. This solution should be standardized at least once every week. It will be found more convenient to make a solution as above described and standardize it every week, than to attempt, by boiling and filtering, to make a solution which will not change.

28. Sodium oxalate for standardization of potassium permanganate solution should be purchased from the U. S. Bureau of Standards, Washington, D. C. This material is sold by the Bureau of Standards as Sample No. 40a.

29. When the end point is reached, the temperature of the solution should not be below 60° C.

TENTATIVE METHODS OF SAMPLING, INSPECTION, PACKING AND MARKING OF QUICKLIME AND LIME PRODUCTS[1]

Serial Designation: C 50 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1922; REVISED, 1923, 1924.

I. METHODS OF SAMPLING

A. General

General. 1. (*a*) Samples of quicklime and lime products shall be taken at one of the following places, as may be agreed upon by the contracting parties:

At point of manufacture, or destination: From conveyors delivering to bins or piles, from filled bins by means of sampling tubes, from filled bins at the point of discharge, from piles by means of sampling tubes or shovels, or from loaded cars.

(*b*) All samples shall be taken in triplicate and immediately sealed in air-tight moisture-proof containers.

(*c*) One sample shall be immediately delivered to the consignee; one sample shall be delivered to the consignor if requested within 10 days from the date of sampling; the third sample shall be retained with seal unbroken until such time as the material has been accepted or a referee test called for.

(*d*) Samples shall be of an equal weight of not less than 5 lb. each when the quantity sampled equals 30 tons or more. For smaller quantities, the weights of the samples may be reduced proportionately, provided that no sample shall weigh less than 2 lb., except by special agreement of the contracting parties.

(*e*) If the samples are taken at any place other than the place of manufacture of the material being sampled, such samples shall be taken within 24 hours of the receipt of the material.

(*f*) Sampling shall be conducted as expeditiously as possible in order to avoid undue exposure of the material to the air.

(*g*) Samples shall not be taken from the broken packages.

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

B. *Lump or Granular Material*

2. (*a*) *In Bulk* (Piles or cars).—Material shall be selected so that it will represent an average of all parts of the pile or car and shall not contain a disproportionate share of the top or bottom layers. This material shall comprise at least 10 shovelfuls taken from different parts of the pile or car. The total material so taken shall weigh not less than 100 lb. for a 30-ton unit, with a proportionate quantity of material for multiples of this unit, provided that at no time shall the total amount of material so taken weigh less than 50 lb. It shall be broken, if necessary, to pass a 1-in. ring, thoroughly mixed, and quartered, and the triplicate samples for testing shall be taken from the material so drawn and prepared. **Lump or Granular Material in Bulk.**

(*b*) *In Bulk* (At conveyors to or from bins, piles or cars).—Material shall be taken at regular intervals during the entire time of movement of the mass being sampled. The quantity to be taken at each interval and the number of such intervals shall be so proportioned that the final quantity of material taken shall conform to the requirements of Paragraph (*a*).

3. *In Packages.*—At least 2 per cent of the packages shall be sampled, the packages to be taken from various parts of the material being sampled, and at no time shall less than 5 packages be sampled. The contents of each of the packages so taken shall be put in a pile, broken, if necessary, to pass a 1-in. ring, thoroughly mixed, and quartered, and the triplicate samples for testing shall be taken from the material so drawn and prepared. **Lump or Granular Material in Packages.**

C. *Powdered Material*

4. (*a*) *In Bulk* (Bins, piles or cars).—A sampling tube shall be used which takes a core not less than 1 in. in diameter, and which is of sufficient length to permit the taking of a core from the top to the bottom of the mass being sampled. Cores shall be taken from at least 10 separate parts of the mass. The cores so taken shall be thoroughly mixed and quartered and the triplicate samples for testing shall be taken from the material so drawn and prepared. A minimum quantity of 100 lb. shall be drawn for a unit of 30 tons of material with a proportionate quantity of material for multiples of this unit, provided that at no time shall less than 50 lb. be drawn as the original portion. **Powdered Material in Bulk.**

(*b*) *In Bulk* (At conveyors to or from bins, piles or cars, at packing machines, etc.)—Sampling at conveyors, packing machines, etc., shall conform to the provisions of Section 2 (*b*).

Powdered Material in Packages.

5. *In packages.*—A sampling tube shall be used which takes a core not less than ½ in. in diameter. The tube shall be so inserted into the package that it will take a core of the material being sampled from substantially the entire length of the package. Material thus taken from individual packages shall be thoroughly mixed and quartered, and the triplicate samples for testing shall be taken from the material so drawn and prepared. Individual packages shall be taken from various parts of the unit being sampled. At least 1.5 per cent of the packages shall be sampled, and in no case shall less than 5 packages be sampled.

D. *Treatment of Sample at Laboratory*

Sample for Chemical Analysis.

6. The sample as received at the laboratory shall be thoroughly mixed, and reduced in quantity by the usual methods of quartering to give a minimum portion of 75 g. This portion shall be ground, either by a suitable mechanical device, or by hand in an agate mortar, until it all passes a No. 100 sieve. It shall then be thoroughly mixed, placed in an air-tight container, and used in the determination of the chemical composition.

Samples for Special Tests.

7. Samples to be used for such special tests as may be required by individual specifications shall be obtained by mixing and quartering the material remaining after removal of the 75-g. portion for chemical analysis. The quantity of sample and the screen size of the particles will depend upon the requirements of the individual specifications.

II. INSPECTION

Inspection.

8. When inspection and sampling are to be conducted at the point of manufacture, the manufacturer shall afford the inspector representing the purchaser all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the operation of the works. The duties of the inspector or sampler shall be confined exclusively to sampling and inspection of the finished material as ready for shipment, and shall not under any circumstances be construed to permit investigation of methods or processes of manufacture.

III. REJECTION

Rejection.

9. (*a*) Rejection of material based on failure to pass the tests prescribed in the specifications shall be reported within 10 days from the taking of samples, and the cause for rejection shall be stated.

(*b*) The samples which represent rejected material shall be kept in air-tight, moisture-proof containers for at least 5 days from the date of the original test report.

IV. RETESTING

10. (*a*) Either of the contracting parties may make claim for a retest within five days of the date of the original test report. The expense of the retest shall be borne by the party demanding such retest. **Retesting.**

(*b*). Should the contracting parties be unable to reach a mutually satisfactory agreement based upon the results of the original test, the third sample of material shall be delivered unopened to a referee laboratory for test and the results of this referee test shall be binding on both parties.

V. PACKING

11. *Lump Lime.*—Lump lime may be shipped in bulk in carload lots, in wooden barrels or steel containers holding 180 lb. or 280 lb. net each, or in pasteboard cartons holding 90 lb. net each. **Packing.**

12. *Pulverized Lime.*—Pulverized lime may be shipped in bulk, in carload lots, or in cloth or paper bags.

13. *Hydrated Lime.*—Hydrated lime shall be shipped in cloth bags holding 100 lb. net each, or in paper bags holding 50 lb. net each.

VI. MARKING

14. Each shipment of material shall carry with it some means of identification, stating the particular contract on which the purchase and shipment were made. **Marking.**

15. Each package shall have legibly marked thereon, the net weight of its contents, the name of the manufacturer, the place of manufacture, and the brand name, if any. This marking shall comply with all requirements of the Federal Lime Barrel Act, entitled "An Act to Standardize Lime Barrels (Public No. 228-Congress-S 5425)," and with such other federal or state laws as may be applicable to the shipment in question.

16. In addition to the above information, the following guarantee may be marked on each package or shipment: "Guaranteed by (name of manufacturer) to meet the specifications of the American Society for Testing Materials" for (name of material).

TENTATIVE METHOD OF TEST

FOR

RESISTANCE OF FIRE-CLAY BRICK TO SPALLING ACTION[1]

Serial Designation: C 38 – 21 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921.

Object. 1. The object of this test is to determine the resistance of high-duty fire-clay brick to spalling action, by subjecting them to repeated rapid temperature changes.

Preparation of Samples. 2. (*a*) The sample shall consist of at least five standard 9-in. brick or of five shapes, which may be cut to standard size with a grinding wheel.

(*b*) Test specimens of shapes such as boiler arch brick shall be cut to the standard 9-in. size. For this purpose a "cut-off" wheel is recommended.

(*c*) The test specimens shall be marked for identification and then subjected to a preliminary heat treatment by heating them uniformly in a suitable furnace to a temperature of 1400° C. (2552° F.), maintaining this temperature for five hours, and then allowing them to cool.

Procedure. 3. (*a*) The specimens shall be weighed and placed in a door of a furnace operated at 1350° C. (2462° F.), care being taken to prevent the direct heating of more than the $2\frac{1}{2}$ by $4\frac{1}{2}$-in. end. After one hour the specimen shall be withdrawn from the furnace and stood on end in a tank of flowing cold water from 10 to 21° C. (50 to 70° F.) so that the hot end of the brick will be immersed to a depth of 4 in. After three minutes the sample shall be withdrawn from the water, allowed to steam five minutes and then returned to the furnace. The door of the furnace shall be closed while the bricks are out to prevent the lowering of the temperature. The alternate heating and cooling shall be continued in hourly cycles until the end of the brick spalls off.

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. F. A. Harvey, Secretary of Committee C-8 on Refractories, United States Refractories Corporation, Mt. Union, Pa.

(*b*) When pieces begin to fall off, the sample is laid on a 4.5 by 9.0-in. asbestos board, divided into one hundred equal squares, and the approximate percentage of loss estimated after each removal from the water. After the completion of the test, the brick is again weighed and the percentage of loss determined.

(*c*) As an alternate for water dipping, air cooling may be used. This method should be used where the spalling is too rapid to get comparative results by the water dipping method. In this case the hot end of the brick shall be placed facing the air blast, 4 in. from the end of a 2-in. pipe from a $\frac{1}{4}$-h.p. blower, delivering between 28 and 30 cu. ft. of air per minute.

4. The report shall show the number of dips before loss started, the percentage of loss after each cooling, the total number of dips and the final percentage of loss by weight. **Reporting Results.**

TENTATIVE METHOD

OF

SECURING SPECIMENS OF HARDENED CONCRETE FROM THE STRUCTURE[1]

Serial Designation: C 42 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1925.

Scope.

1. The methods and precautions herein specified apply to securing test specimens from hardened concrete in the structure.

Precautions.

2. A specimen from hardened concrete to be tested for strength shall not be taken until the concrete has become hard enough so that the cutting of the specimen will not disturb the bond between the mortar content and the coarse aggregate of the specimen.

Apparatus.

3. The specimen from the structure can best be secured by use of a core drill. For specimens taken perpendicular to a horizontal surface, a drill using chilled shot may be used; but when taken perpendicular to a vertical surface, a diamond drill should be used.

Test Specimen.

4. (*a*) The core specimen taken shall be as nearly as possible a cylinder whose length is twice the diameter.

(*b*) In securing a specimen perpendicular to a horizontal surface care shall be taken to secure, if possible, a specimen whose beds shall be parallel to the horizontal bed of the concrete as originally placed.

(*c*) In securing a specimen perpendicular to a vertical surface, or to a surface with a batter, care shall be taken as to the point of securing the specimen. The lower portion of any one unit of depositing in mass concrete is more dense than is the upper portion of the unit, therefore a specimen shall be taken from near the middle of such unit of deposit.

Procedure.

4. A specimen to be taken from hardened concrete which has been removed from a structure shall be cut out by a drill or shall be cut into a symmetrical test-piece by tooling or by sawing. The method of sawing can seldom be used to advantage in the field. In selecting

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. J. C. Pearson, Secretary of Committee C-9 on Concrete and Concrete Aggregates, Lehigh Portland Cement Co., Allentown, Pa.

this test specimen care shall be used to see that the concrete selected has not been injured or shattered by the method of its removal from the structure.

Ends.

5. The specimens secured shall have ends, or beds, as nearly as possible perpendicular to its axis, and, if the specimen has ends with uneven surfaces, these ends shall be made to parallel plane surfaces with a mortar richer than the mortar of the specimen or with a mixture of cement and calcined gypsum (plaster of Paris).

6. In case the dimensions of a specimen are necessarily appreciably different from the recommended shape which requires a height twice the diameter, allowance for the ratio of height to diameter must be made in determining the strength. When the height is more than twice the diameter, that is, when the ratio is greater than 2, no correction need be made. For other ratios corrections should be made as follows:

Ratio of Height of Cylinder To Diameter, $\frac{h}{d}$	
1.75	multiply crushing strength by 0.98
1.50	" " " " 0.95
1.25	" " " " 0.94
1.10	" " " " 0.90
1.00	" " " " 0.85
0.75	" " " " 0.70
0.50	" " " " 0.50

The direction of the application of the load in the compression test shall be stated with reference to the direction of the compaction of the mass of concrete in the structure, and where it is practicable the compression load shall be applied in the same direction. In order that the tests be made under uniform conditions as to moisture content, the test specimens shall be completely submerged in water for 48 hours and the compression test shall be made immediately thereafter.

TENTATIVE DEFINITIONS OF TERMS RELATING TO LIME[1]

Serial Designation: C 51 - 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. **It is not a Standard of the Society and is subject to annual revision.**

ISSUED, 1922; REVISED, 1924.

I.

Quicklime.—A calcined material, the major part of which is calcium oxide or calcium oxide in natural association with a lesser amount of magnesium oxide, capable of slaking with water.

Hydrated Lime.—A dry powder obtained by treating quicklime with water enough to satisfy its chemical affinity under the conditions of its hydration.

NOTE.—It consists essentially of calcium hydroxide or a mixture of calcium hydroxide and magnesium oxide and magnesium hydroxide.

In Bulk.—Lime or lime products are "in bulk" when it or they exist *en masse* in bins, in piles, or in transporting vehicles.

II. SIZES OF QUICKLIME

Lump Lime.—Quicklime as it comes from the kilns.

Lump Lime Screened.—Lump lime after forking or screening to remove the portion which will pass a $\frac{1}{2}$-in. sieve.

Pulverized Lime.—Quicklime which will pass a $\frac{1}{4}$-in. sieve.

[1] Criticisms of these Tentative Definitions are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

TENTATIVE DEFINITIONS OF TERMS RELATING TO THE GYPSUM INDUSTRY[1]

Serial Designation: C 11 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1916; Revised, 1921, 1922, 1923, 1924, 1925.

Accelerator.—Any material or substance which, when used in stucco, plaster, mortar, concrete, etc., will hasten set beyond the limit of testing error.

Aggregate.—Any inert material used as a filler in stucco, plaster, mortar, concrete, etc., without regard to its function as a binding material.

Binder.—Any fibrous material which will increase the cohesiveness of plaster and stucco while in its plastic state.

Cement.—Any material, or a mixture of materials (without aggregate) which becomes plastic upon the addition of water and, when in a plastic state, possesses adhesive and cohesive properties, and hardens in place. The word "cement" is used without regard to the composition of the material and does not define its use or location of use as do the words "stucco," "plaster" and "mortar."

Consistency.—The degree of wetness of a plastic mixture.

Calcined Gypsum.—Gypsum partially dehydrated by means of heat, having the chemical formula $CaSO_4 \cdot \frac{1}{2}H_2O$.

Crushed Gypsum.—Gypsum subjected to a primary crushing operation.

Gypsum.—Calcium sulfate, combined with two molecules of water, in crystallin form, having the chemical formula $CaSO_4 \cdot 2H_2O$.

Gypsum Plastering Sand.—The fine granular material naturally or artificially produced by the disintegration of rock containing not less than 80 per cent by weight of silica, feldspar, dolomite, magnesite or calcite.

Gypsum Plaster Board.—A sheet composed of an incombustible core of gypsum, surfaced with paper or other fibrous material which is firmly bonded to the core, or with intermediate layers of such material within the core, or an incombustible sheet of

[1] Criticisms of these Tentative Definitions are solicited and should be directed to Mr. V. G. Marani, Secretary of Committee C-11 on Gypsum, 844 Rush St., Chicago, Ill.

gypsum with not more than 15 per cent by weight of fiber intimately mixed; and designed to be used as a lath for the reception of gypsum plaster.

Gypsum Wall Board.—A sheet composed of an incombustible core of gypsum, surfaced with paper or other fibrous material which is firmly bonded to the core, and designed to be used, without the addition of plaster, for walls, ceilings or partitions and affording a surface suitable to receive decoration.

Lath.—A material whose primary function is that of a base or background for the reception of plaster or stucco. The word "lath" is used without regard to the composition of the material, defining only its use.

Mortar.—Any material used in a plastic state that can be trowelled, and becomes hard in place, uniting masonry materials. The word "mortar" is used without regard to the composition of the material, defining its use as a bonding material, as contrasted with the words "stucco" and "plaster."

Plaster.—Any material used in a plastic state to form a hard covering for the interior surfaces, walls and ceilings, etc., of any building or structure. The word "plaster" is used without regard to the composition of the material, defining only its use and location of use, as contrasted with the words "stucco" and "mortar."

Plasticity.—The yielding property of a wet mixture to change of form.

Retarder.—Any material or substance which, when used in stucco, plaster, mortar, concrete, etc., will retard set beyond the limit of testing error.

Sized Gypsum.—Crushed gypsum of prescribed diameter of individual particles.

Stucco.—Any material used in a plastic state to form a hard covering for the exterior walls or other exterior surfaces of any building or structure. The word "stucco" is used without regard to the composition of the material, defining only its use and location of use, as contrasted with the words "plaster" and "mortar."

Wood Fiber.—A material produced by grinding or shredding wood.

TENTATIVE DEFINITION OF THE TERM SAND[1]

Serial Designation: C 58-25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924; Revised, 1925.

Sand.—The fine granular material resulting from the natural disintegration or the crushing of rock or slag.

Note.—When used without a qualifying adjective it is generally understood to mean a product of the natural disintegration of siliceous or calcareous rock. The size of particle and other physical characteristics should be taken care of in specifications and therefore no limits are mentioned in the definition.

[1] Criticisms of this Tentative Definition are solicited and should be directed to Mr. L. R. Ferguson, Chairman of the Sub-Committee, of Committee E-8, on Sand, International Cement Corporation, 342 Madison Ave., New York City.

TENTATIVE RULES

FOR

INSPECTION OF CONCRETE AND REINFORCED CONCRETE WORK.[1]

Serial Designation: C 44 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922.

Committee C-2 on Reinforced Concrete in presenting these Tentative Rules for Inspection of Concrete and Reinforced Concrete Work submits, as a guide in selecting the inspector, its recommendations as to his qualifications. These recommendations cannot, in fact, form a part of the rules.

QUALIFICATIONS OF AN INSPECTOR.

The inspector should have practical experience and an understanding of the fundamental engineering principles involved in the construction to which he is assigned; he preferably should have had technical training. The inspector should keep abreast of developments by reading the engineering and trade press, books and monographs on the subject and by attendance at meetings at which concrete work is discussed. Concrete and reinforced concrete construction is a rapidly-developing art and it is imperative that the inspector should be informed as to the most recent improvements, if he is to handle his work intelligently and efficiently.

STATUS OF INSPECTOR.

1. The word inspector as hereinafter used is a generic term. The inspector is the authorized representative of the engineer who is the authorized representative of the owner.

NOTE.—A grave responsibility rests on the inspector. The success of the construction may hinge on the proper performance of what may appear to be minor features of the work. In case of careless or faulty construction which results in loss of life the inspector may be charged with criminal negligence.

[1] Criticisms of these Tentative Rules are solicited and should be directed, preferably before January 1, 1926, to Mr. Richard L. Humphrey, Chairman of Committee C-2 on Reinforced Concrete, 805 Harrison Building, Philadelphia, Pa.

RESPONSIBILITIES AND GENERAL DUTIES OF INSPECTOR.

2. It is the duty of the inspector to see that the work is executed in full accordance with the plans and specifications.

3. The inspector will be provided with complete plans and specifications for the work and shall familiarize himself with all their requirements.

NOTE.—The inspector should read and check off all dimensions and notes on the plans and list all special requirements in connection with the work to which they apply; when in doubt he should obtain instructions sufficiently in advance of the work to avoid delay.

4. The inspector shall familiarize himself with the Building Code or Regulations under which the work is being done, including all special rulings, which have a bearing on the type of construction under way.

NOTE.—The inspector should be familiar with auxiliary specifications and the common tests for suitability of the materials used in concrete and reinforced concrete work, such as:

(*a*) Standard Specifications and Tests for Portland Cement.
(*b*) Standard Specifications for Concrete Reinforcement Bars, etc.

5. The inspector shall be on the work at all times when it is in progress.

6. The inspector shall not permit work to proceed until requisite lines and levels have been established.

7. The inspector is authorized to:

(*a*) Forbid use of materials or workmanship which do not conform to specifications;
(*b*) Stop any work which is not being done in accordance with the plans and specifications;
(*c*) Require, with the approval of the engineer, the removal or repair of faulty construction.

8. The inspector shall immediately report to his immediate superior any action taken by him under Rule 7.

9. The inspector shall report at once to his immediate superior any discrepancies in plans or specifications that may come to his attention and request instructions.

10. The inspector shall not permit any departure from plans or specifications without written instructions from his immediate superior.

11. The inspector shall record and report daily the following information:

(*a*) Date;
(*b*) Location of work under way;
(*c*) Name of contractor;

(*d*) Delivery, installation, and removal of contractor's equipment;
(*e*) Number and classification of laborers, teams, plant, etc., in use;
(*f*) Materials received and on hand;
(*g*) Inspection and location of forms erected and completed reinforcement in place;
(*h*) Location and approximate quantities of concrete placed and bags of cement used;
(*i*) Time of commencement and completion of placing concrete;
(*j*) Number and location of samples taken;
(*k*) Nature and time of any control tests made;
(*l*) Forms removed;
(*m*) All items on force account work;
(*n*) Weather conditions, and temperature readings taken morning, noon and evening, including also the maximum and minimum temperatures during each preceding period;
(*o*) Material rejected or work condemned under Rule 7;
(*p*) Disposition of rejected materials;
(*q*) Any special instructions given to contractor;
(*r*) Any unusual features of the day's work;
(*s*) Signature of inspector.

NOTE.—It is not expected that the inspector shall act as time-keeper or materials clerk, but will record the data called for and, so far as possible, indicate the source of the information. For convenience, the inspector should be supplied with printed forms for daily reports.

12. The inspector shall procure, at the expense of the owner, photographs taken at frequent intervals to show the progress of the work.

13. The inspector, on force account work, shall check over with the authorized representative of the contractor, at the close of each day's work, all items included therein.

NOTE.—It is desirable to have printed forms on which the above can be reported daily and signed by the inspector and by the contractor's authorized representative.

EXCAVATION AND FOUNDATION.

14. The inspector shall examine the finished excavation and determine whether it is down to the level specified and see that the material on which the foundation is to rest is in accordance with the plans and specifications. If excavation is to go to rock he shall see that the surface of the rock is sound and completely exposed. He shall secure the approval of the engineer before permitting concrete to be placed.

15. The inspector shall see that the required number of piles are driven, each in its proper location and to the penetration required by the specifications.

16. The inspector shall check the piles in each group after driving and should report any pile which is materially out of line. He shall keep a record of:

(*a*) Method of driving;
(*b*) Type of pile;
(*c*) Length of pile;
(*d*) Diameter of pile;
(*e*) Total depth of penetration;
(*f*) Type and weight of hammer;
(*g*) Drop of hammer under last five blows;
(*h*) Penetration of pile under last five blows;
(*i*) Number of piles driven;
(*j*) Number of piles specified;
(*k*) Length of pile under cutoff.

17. The inspector shall see that there are no pockets in the concrete and that excessive laitance is removed.

18. The inspector shall see that the requirements of the specifications for placing concrete under water are strictly followed. In the absence of specifications, standard methods shall be used.

NOTE.—In placing concrete under water it is highly important that extra precautions be taken by the contractor to execute the work properly in order that the concrete may have adequate density, be free from segregations, or the washing out of any of the materials, and to avoid exposure of freshly deposited concrete to the water.

MATERIALS.

19. The inspector shall:

(*a*) Inspect and identify each lot of material upon its receipt on the work;
(*b*) Keep a careful watch of aggregates, especially if the source of supply is changed from time to time;
(*c*) See that aggregates are clean and properly graded.
(*d*) See that aggregates are free from vegetable or animal matter as determined by the colorimetric test described in the Standard Method of Test for Organic Impurities in Sands for Concrete (Serial Designation: C 40) of the American Society for Testing Materials.[1]
(*e*) See that materials are properly stored on the work.

[1] 1924 Book of A.S.T.M. Standards.

NOTE.—The cement should be properly piled and tagged; and housed in such a manner as to protect it from the weather.

Aggregates should be piled in such a manner as to insure them against intermingling of foreign materials.

Metal reinforcement should be stored preferably under shelter.

20. The inspector shall observe the following in selecting samples where tests of concrete materials are required under the specifications:

(*a*) Secure at least a 20-lb. sample of fine aggregate;

(*b*) Secure at least a 50-lb. sample of coarse aggregate;

(*c*) Give complete information concerning the sample (both on the inside and on the outside of the package);

(*d*) Samples shall be shipped in a strong sack or tight box; if a sack is used it shall be clean.

NOTE.—Tests on materials are worthless unless sampling is *properly* done. Particular care must be used in sampling aggregates under the following conditions:

(*a*) Aggregates in undeveloped pits;

(*b*) Coarse aggregate in cars or stock piles;

(*c*) Rock in native ledges.

NOTE.—The method of selecting test samples shall be as described in the Standard Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (Serial Designation: D 75) of the American Society for Testing Materials.[1]

METAL REINFORCEMENT.

21. The inspector shall see that the metal reinforcement, before being positioned, is cleaned of mill and rust scale, and of coatings of any character that may destroy or reduce the bond. He shall reject reinforcement appreciably reduced in section. If there is delay in depositing concrete he shall re-inspect the reinforcement and when necessary shall see that it is cleaned.

22. The inspector shall see that the required bending of bars is uniformly and accurately done.

NOTE.—Bends can be more uniformly and accurately made by machine than by hand. In case a considerable number of bars are to be bent alike, it is advisable to bend one, put it in position and check the bends before bending the others, to insure proper location and degree of bends.

23. The inspector shall see that the reinforcement is of the specified size, bent in accordance with the specifications, and properly positioned in the forms before placing the concrete.

NOTE.—The inspector should not wait until the metal reinforcement has been wired substantially in place before passing on the correctness of its position, but should inspect the reinforcement during its placing.

[1] 1924 Book of A.S.T.M. Standards.

The inspector should inform himself as to the details for the placing of the reinforcement, especially as to size, length, bending, laps, and splices, and in case of doubt should secure instructions.

24. The inspector shall see that the bars are rigidly secured and that they are not displaced during the placing of the concrete.

25. The inspector shall see that all joints and splices in metal reinforcement are located and made in full accordance with the specifications.

MIXING AND PLACING OF CONCRETE.

26. The inspector, in order to secure the proportions and mixing required by the specifications, shall:

(*a*) Check the capacity of wheelbarrows or other measuring devices, and see that the method of loading them will secure uniformly the specified proportions;

(*b*) Record total quantities of cement and aggregates used;

(*c*) See that the specified consistency is maintained throughout the work;

(*d*) See that the time of mixing is that specified;

(*e*) See that changes in proportions and consistency, ordered by his immediate superior, are carried out;

(*f*) See that the samples required for tests of concrete are taken as provided in the Standard Method of Making and Storing Specimens of Concrete in the Field (Serial Designation: C 31) of the American Society for Testing Materials.[1]

27. The inspector shall see that the concrete is placed in accordance with the specifications, and specifically that:

(*a*) The method of conveying the concrete is as specified; or in the absence of specifications that it is such as to avoid segregation or the formation of excessive laitance;

(*b*) The concrete is placed in the form as near as possible to the point of its final location and is not permitted to flow long distances in the form;

(*c*) It is properly spaded or tamped so as to fill the forms and surround the reinforcement;

(*d*) It is properly spaded or forked to keep the coarse aggregate away from the forms so as to insure a smooth surface;

[1] 1924 Book of A.S.T.M. Standards.

(*e*) The concrete is placed continuously without undue delay in each unit of the structure;

(*f*) Construction joints are made in accordance with the specifications; in the absence of specifications, that written instructions are obtained from his immediate superior.

(*g*) The concrete is poured in a continuous operation between construction joints;

(*h*) Where night work is necessary, adequate precautions as to lighting, etc., are taken;

(*i*) All anchor bolts, inserts, pipe sleeves, wiring, drainage pipes, flashings, conduits and other fixtures are in position as required by the plans and specifications before the concrete is placed; and that conduits and other equipment are built into the slab in such a manner as not to affect the position of metal reinforcement or weaken the member at critical sections;

(*j*) The specified precautions are strictly observed when it is necessary to place concrete in cold weather;

(*k*) A prompt report is made where conditions require additional fittings or where installations not called for in the specifications should be in position before the concrete is placed.

28. The inspector shall see that the freshly-placed concrete is protected and kept moist in accordance with the specifications.

NOTE.—Proper curing of the concrete is particularly important in floors or stairs where the concrete is not entirely covered by forms. This single precaution may add as much as 50 per cent to the crushing strength of concrete, greatly increase resistance to wear, and prevent "dusting" of floors.

FORMS.

29. The inspector shall examine the forms before placing concrete and:

(*a*) Check same as to location and dimensions:

(*b*) See that the surface of the form is oiled or wetted in accordance with the specifications;

(*c*) Report immediately any seeming lack of strength in forms which would permit bulging or sagging between supports;

NOTE.—This pressure amounts to over 100 lb. per sq. ft. of surface for each foot of depth, where the form is filled in forty minutes or less. It is very difficult and usually impossible with the equipment available to force a form back into position after it has shifted or sagged out of place while being filled.

(*d*) See that the column and girder forms are straight and properly aligned;

(*e*) See that foreign materials, such as chips, blocks, and shavings are removed from forms before concrete is placed.

REMOVAL OF FORMS.

30. The inspector shall see that the forms are removed:

(*a*) At the time and in the manner required;

(*b*) With care so as not to injure the structure or mar the surface of the concrete.

31. The inspector shall see that:

(*a*) The forms are not removed until the concrete has hardened sufficiently for safety;

(*b*) Special precautions are taken to protect the concrete during cold weather;

NOTE.—Concrete hardens very slowly at a temperature below 50° F. and setting action is scarcely perceptible at temperatures below 40° F. The time during which the concrete has been exposed to temperatures below 40° F. should be added to the periods stated in the schedule in determining the time of removal of forms. The effect of cold weather on the strength of concrete is well illustrated in *Bulletin 81* of the Engineering Experiment Station of the University of Illinois on "Influence of Temperature on the Strength of Concrete," by A. B. McDaniel.

(*c*) Any uncertainty as to the conditions affecting the removal of forms is promptly reported to his immediate superior;

(*d*) Defects in the surface of the concrete are repaired promptly upon the removal of forms;

(*e*) Heavy loading on green concrete is not permitted.

TENTATIVE SPECIFICATIONS

FOR

RAW TUNG OIL[1]

Serial Designation: D 12 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923, 1925.

I. PROPERTIES AND TESTS.

Properties. 1. Raw tung oil shall conform to the following requirements:

	MAXIMUM.	MINIMUM.
Specific gravity at 15.5°/15.5° C	0.943	0.9400
Acid number (Alcohol-Benzol)	8.0	.....
Saponification number	195	190
Unsaponifiable matter, per cent	0.75	.....
Refractive index at 25° C	1.520	1.5165
Iodine number (Wijs)	.....	163
Heating test, minutes	12	.....

II. METHODS OF TESTING.

SOLUTIONS REQUIRED.

Solutions Required. 2. The following reagents will be required:

Standard Sodium Thiosulfate Solution.—Dissolve pure sodium thiosulfate in distilled water that has been well boiled to free it from carbon dioxide in the proportion so that 24.83 g. crystallized sodium thiosulfate will be present in 1000 cc. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Treadwell-Hall, *Analytical Chemistry*, Vol. II, third edition, p. 646.) This solution will be approximately N/10 and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly N/10 strength. Preserve in a stock bottle with a guard tube filled with soda lime.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

These Tentative Specifications are in effect a revision of the Standard Specifications for Purity of Raw Tung Oil. The Standard Specifications, which were last published under the Serial Designation: D 12 – 16, have accordingly been withdrawn.

Starch Solution.—Stir up 2 to 3 g. of potato starch or 5 g. soluble starch with 100 cc. of 1-per-cent salicylic acid solution, add 300 to 400 cc. boiling water, and boil the mixture until the starch is practically dissolved. Dilute to 1 liter.

Potassium Iodide Solution.—Dissolve 150 g. of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

Iodine-Monochloride Solution.—Dissolve iodine in glacial acetic acid that has a melting point of 14.7 to 15° C. and is free from reducing impurities in the proportion so that 13 g. of iodine will be present in 1000 cc. of the solution. The preparation of the iodine-monochloride solution presents no great difficulty, but it shall be done with care and accuracy in order to obtain satisfactory results. There shall be in the solution no sensible excess either of iodine or more particularly of chlorine, over that required to form the monochloride. This condition is most satisfactorily attained by dissolving in the whole of the acetic acid to be used the requisite quantity of iodine, using a gentle heat to assist the solution, if it is found necessary. Set aside a small portion of this solution, while pure, and pass dry chlorine into the remainder until the halogen content of the whole solution is doubled. Ordinarily it will be found that by passing the chlorine into the main part of the solution until the characteristic color of free iodine has just been discharged, there will be a slight excess of chlorine, which is corrected by the addition of the requisite amount of the unchlorinated portion until all free chlorine has been destroyed. A slight excess of iodine does little or no harm, but excess of chlorine must be avoided.

Chloroform.—Should be U. S. P.

Standard Sodium Hydroxide Solution.—Prepare a stock concentrated solution of sodium hydroxide by dissolving NaOH in water in the proportion of 200 g. NaOH to 200 cc. water. Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the precipitate of sodium carbonate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off about 175 cc. and dilute to 10 liters with freshly boiled distilled water. Preserve in a stock bottle provided with a large guard tube filled with soda lime. Determine the exact strength by titrating against pure benzoic acid (C_6H_5COOH) using phenolphthalein as indicator. (See Bureau of Standards *Scientific Paper No. 183.*) This solution will be approximately N/4, but do not attempt to adjust it to any exact value. Determine its exact strength and make proper corrections in using it.

Alcoholic Sodium Hydroxide Solution.—Dissolve pure NaOH in 95-per-cent ethyl alcohol in the proportion of about 22 g. per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle, and keep well stoppered. This solution should be colorless or only slightly yellow when used; it will keep colorless longer if the alcohol is previously treated with NaOH (about 80 g. to 1000 cc.), kept at about 50° C. for 15 days, and then distilled. For an alternate method see *Journal*, American Chemical Society, 1906, p. 395.

Half Normal Sulfuric Acid Solution.—Add about 15 cc. H_2SO_4 (sp. gr. 1.84) to distilled water, cool and dilute to 1000 cc. Determine the exact strength by titrating against freshly standardized NaOH or by any other accurate method. Either adjust to exactly N/2 strength or leave as originally made, applying appropriate correction.

METHODS.

Methods of Testing.

3. The oil shall be tested in accordance with the following methods:

General.—The laboratory sample shall be thoroughly mixed by shaking, stirring, or pouring from one vessel to another and the samples for the individual tests taken from this thoroughly mixed sample.

Specific Gravity.—Use a pyknometer accurately standardized and having a capacity of at least 25 cc., or any other equally accurate method, making the test at 15.5° C., water being 1 at 15.5° C.

Acid Number.—Weigh from 5 to 10 g. of the oil. Transfer to a 300-cc. Erlenmeyer flask. Add 50 cc. of a mixture of equal parts by volume of 95-per-cent ethyl alcohol and c. p. reagent benzol. (*This mixture should be previously titrated to a very faint pink with dilute alkali solution, using phenolphthalein as an indicator*). Add phenolphthalein indicator and titrate at once to a faint permanent pink color with the standard sodium hydroxide solution. Calculate the acid number (milligrams KOH per gram of oil).

Saponification Number.—Weigh about 2 g. of the oil in a 300-cc. Erlenmeyer flask. Add 25 cc. alcoholic sodium hydroxide solution. Put a condenser loop inside the neck of the flask and heat on the steam bath for one hour. Cool, add phenolphthalein as indicator, and titrate with N/2 H_2SO_4. Run two blanks with the alcoholic sodium hydroxide solution. These should check within 0.1 cc. N/2 H_2SO_4. From the difference between the number of cubic centimeters of N/2 H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams KOH required for 1 g. of the oil).

Unsaponifiable Matter.—Weigh 8 to 10 g. of the oil. Transfer to a 250-cc. long-neck flask. Add 5 cc. of strong solution of sodium hydroxide (equal weights of NaOH and H_2O), and 50 cc. 95-per-cent

ethyl alcohol. Put a condenser loop inside the neck of the flask and boil for two hours. Occasionally agitate the flask to break up the liquid but do not project the liquid onto the sides of the flask. At the end of two hours remove the condenser and allow the liquid to boil down to about 25 cc.

Transfer to a 500-cc. glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 cc., add 100 cc. redistilled ether. Stopper and shake for one minute. Let stand until the two layers separate sharp and clear. Draw all but one or two drops of the aqueous layer into a second 500-cc. separatory funnel and repeat the process, using 60 cc. of ether. After thorough separation draw off the aqueous solution into a 400-cc. beaker, then the ether solution into the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the seond separatory funnel and shake out again with 60 cc. of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

Shake the combined ether solution with the accumulated water rinsings and let the layers separate sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 cc. each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stopcock, and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw the ether solution (portionwise if necessary) into a 250-cc. flask and distill off. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool and weigh.

NOTE.—The unsaponifiable oil from adulterated drying oils is volatile and will evaporate on long heating. Therefore heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.

Refractive Index.—Use a properly standardized Abbé refractometer at 25° C., or any other equally accurate instrument.

Iodine Number.—Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer by dropping from 0.16 to 0.19 g. to a 500-cc. bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine tests. Reweigh the burette or beaker and determine the amount of sample used. Add 10 cc. of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc. of chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 cc. of the

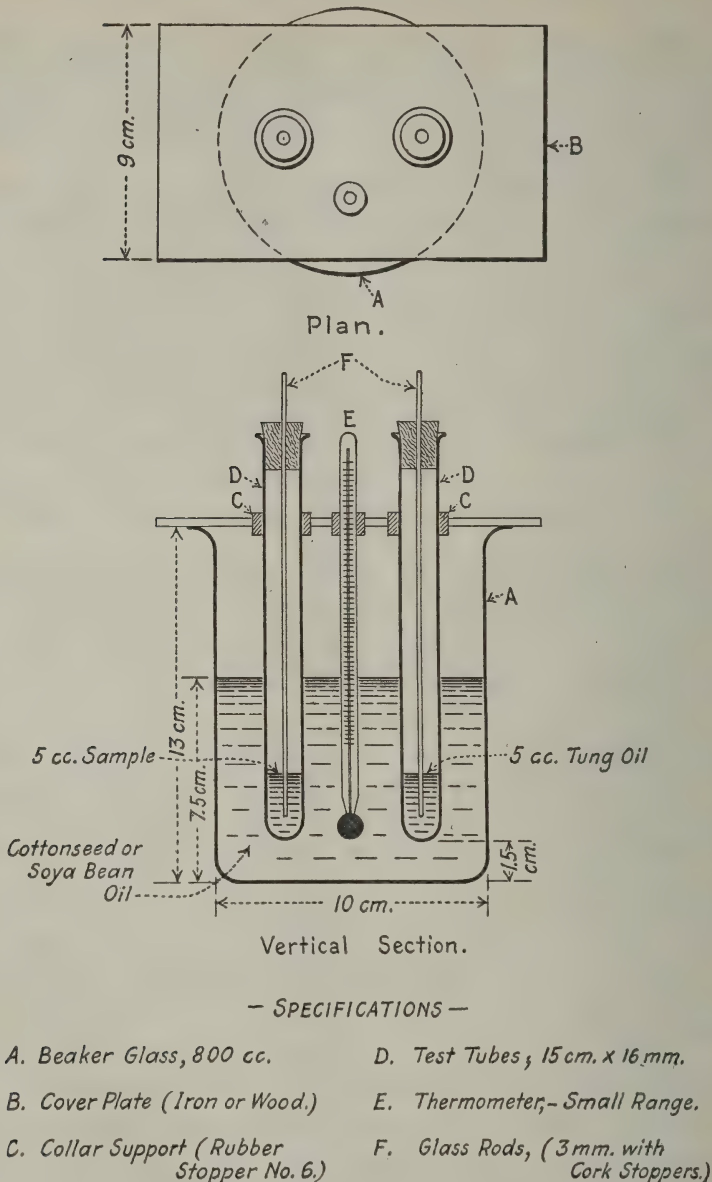

FIG. 1.—Tung Oil Heat Test Apparatus (Revised 1920).

NOTE: Collars *C* may be omitted and tubes supported in present place by aid of wire gauze placed in bottom of oil bath and resting on bottom of beaker.

Wijs solution and let stand with occasional shaking for 30 minutes in a dark place at a temperature of from 21 to 23° C. Add 10 cc. of the 15-per-cent potassium iodide solution and 100 cc. of water, and titrate with standard sodium thiosulfate using starch as the indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titration and the titration on the samples and the iodine value of the thiosulfate solution, calculate the iodine number of the samples tested. (Iodine number is given in centigrams of iodine to 1 g. of sample.)[1]

Heating Test.—Test tubes containing the oil should be 15 cm. by 16 mm., with a mark near the bottom to indicate 5 cc., and closed by a cork so perforated that a glass rod 3 mm. in diameter can move freely.

Fill an 800-cc. glass beaker (height, 13 cm.; diameter, 10 cm.). with cottonseed oil or soya bean oil to a height of 7.5 cm. Place a thermometer so as to be 1.5 cm. from the bottom of the bath. (See Fig. 1.)

Use a nitrogen-filled, chemical thermometer; engraved stem; total length 4 to 4½ in., graduated from 210 to 310° C. in 2° intervals; the length between 210 and 310° C. not less than 2½ in. Thermometer glass shall be well annealed.

When the bath temperature is 293° C. (560° F.) and very slowly rising at this point, place the tube containing 5 cc. of the oil to be tested and the tube containing 5 cc. of a control sample of known value, so that the bottom of each tube is level with the lowest part of the bulb of the thermometer. If desired, the collars *C* may be omitted and the tubes allowed to rest upon a piece of wire gauze placed in the bottom of the oil bath so that the tubes will be 1.5 cm. from the bottom of the bath. Note the time. Remove the source of heat for about 45 seconds and then reapply. Before 2 minutes have elapsed the temperature of the bath will have fallen to 282° C. (530° F.), at which point it should be kept as steady as possible. When the samples have been in the bath 9 minutes, raise the glass rods at intervals of ¼ minute. Note the time when each sample becomes firmly set. At this period the oil will be so stiff that the entire tube may be lifted by aid of the rod if the collar *C* is omitted from the apparatus. As setting or jellying takes place within a few seconds of fluidity, a good end determination is afforded. Remove the specimens. Heat the bath again to 293° C., and repeat the experiment with fresh portions of the sample.

No stirrer is used in the bath. A screen around the bath enables the temperature to be more easily reached. When the bath oil has become tarry and viscid, it should be renewed; otherwise heating may be irregular.

[1] It is always well to include a test on a sample of tung oil of known iodine value. This may be kept in a dark-colored bottle as a standard.

Quality Test.[1]—Into an ordinary agateware casserole, having a bottom diameter of 3 in., weigh 150 g. of the tung oil to be tested, and set the casserole on a wide-flanged tripod having a 3-in. opening. The object of the flange is to prevent super-heating of the sides of the casserole. Heat rapidly with a full Bunsen flame, stirring with a thermometer, until the heat reaches 540° F. (282.2° C.). Turn down the flame and hold the heat as near 540° F. (282.2° C.) as possible, stirring with the thermometer, until on lifting the latter the oil drops with a pronounced string, showing that polymerization has started. The time required after reaching 540° F. (282.2° C.) until the string is noted, is the time of the heat test. For pure tung oils this will not exceed eight minutes. As soon as the oil strings, remove the lamp and the thermometer, and stir with a stiff spatula until the oil is solid. After stringing, a pure tung oil will require not over 40 seconds more to become solid. When solid, allow to stand just one minute, then turn out, upside down, on clean paper and cut with a clean spatula. Pure tung oil gives a gel that is dry, not adhering to the spatula when cut, that is firm, crumbling under pressure of the spatula without sticking, and the cut portions should crumble under pressure like dry bread crumbs. Adulterated tung oil gives a gel that is soft, sticky, and which will not crumble.

III. SAMPLING

The method of sampling given under Method (*a*) below shall be used whenever it is feasible to apply it. To meet conditions when (*a*) is not applicable, method (*b*) or (*c*) shall be used, according to the special conditions that obtain.

(*a*) *During Loading of Tank Cars or Filling of Containers for Shipment.*—

The purchaser's inspector shall draw a sample at the discharge pipe where it enters the receiving vessel or vessels. The total sample shall be not less than 1 gal. per each tank car or 10,000 gal. equivalent, and shall be a composite of small samples of not more than 1 pt. each taken at regular intervals during the entire period of loading or filling.

The sample thus obtained shall be thoroughly mixed, and from this composite sample three portions of not less than 1 qt. each shall be placed in clean, dry glass bottles or tin cans, which must be filled with the sample and securely stoppered with new clean corks or well-fitting metal covers or caps. These shall be sealed and labeled distinctly by the inspector, one delivered to the buyer, one to the seller, and the other held for check in case of dispute.

[1] Furnished by R. S. Worstall.

(*b*) *From Loaded Tank Cars or Other Large Vessels.*—

The total sample shall be not less than 1 gal. per each tank car or 10,000 gal. equivalent, and shall be a composite of numerous small samples of not more than 1 pt. each, taken from the top, bottom, and intermediate points by means of a glass or metal container with removable stopper or top. This device attached to a suitable pole is lowered to various desired depths when the stopper or top is removed and the container allowed to fill. The sample thus obtained shall be handled as in Method (*a*). For large shipments in freighters ranging from 400 to 1000 tons, first draw samples from the top and bottom of both port and starboard sections of the tank. These samples shall be visually examined, and if the general appearance gives indications that the oil is satisfactory, pumping shall be started and 1-pt. samples drawn from a bleeder in the discharge line at least once in every thirty minutes, so that the total mixed sample amounts to as many gallons as there are units of 10,000 gal. in the cargo. These individual samples are composited to make one uniform sample representative of the entire cargo and this is handled as in Method (*a*).

(*c*) *Barrels and Drums.*—

Not less than 10 per cent of the packages in any shipment or delivery of barrels and drums shall be sampled. The packages shall be shaken, rolled, and stirred to thoroughly mix the contents. The samples from the individual containers shall be taken through the bung hole or holes not less than $1\frac{1}{4}$ in. in diameter bored in the head or side for the purpose. The apparatus for drawing the sample shall consist of a glass tube (known as a thief) about 1 in. in diameter and somewhat longer than the length or diameter of the oil container, a conical stopper that will fit the glass tube and is not more than $\frac{1}{2}$ in. in length, fastened to a stiff metal rod not more than $\frac{1}{4}$ in. in diameter and not less than 4 in. longer than the glass tube. The stopper is lowered by the rod until it rests on the bottom of the cask, the tube slipped down slowly over the rod, and finally pressed on the stopper. By holding the tube and the rod, the column of oil can then be removed. This process is repeated until the required amount of sample is obtained, which shall be not less than 1 gal. This is mixed and handled as in Method (*a*). During the winter when the oil is very often in a solid condition, the same procedure should be followed, replacing the thief with a tallow trier, which is essentially a graduated piece of steel tubing semi-circular in cross-section and about 3 ft. long. This is pushed into the oil, turned a few times and withdrawn, thereby removing a solid core of oil.

TENTATIVE SPECIFICATIONS

FOR

SOYA BEAN OIL, RAW OR REFINED[1]

Serial Designation: D 124 – 22 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922.

I. PROPERTIES AND TESTS.

Properties.

1. Soya bean oil, raw or refined, shall conform to the following requirements:

	Maximum.	Minimum.
Foots, per cent	2.5	...
Loss on heating at 105 to 110° C., per cent	0.2	...
Specific gravity at 15.5°/15.5° C	...	0.924
Acid number	5.0	...
Saponification number	...	190
Iodine number (Hanus)	...	128
Unsaponifiable matter, per cent	1.5	...
Color	Not darker than a freshly prepared solution of 1.0 g. potassium bichromate in 100 cc. pure H_2SO_4 (sp. gr. 1.84).	

II. METHODS OF TESTING.

Solutions Required.

Solutions Required.

2. The following reagents will be required:

Acetone that will pass the specification of the United States Pharmacopoeia.

Acid Calcium Chloride Solution.—Saturate with calcium chloride a mixture of 90 parts water and 10 parts HCl (sp. gr. 1.18).

Standard Sodium Thiosulfate Solution.—Dissolve pure sodium thiosulfate in distilled water that has been well boiled to free it from

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

carbon dioxide in the proportion so that 24.83 g. crystallized sodium thiosulfate will be present in 1000 cc. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Treadwell-Hall, *Analytical Chemistry*, Vol. II, third edition, p. 646.) This solution will be approximately N/10 and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly N/10 strength. Preserve in a stock bottle with a guard tube filled with soda lime.

Starch Solution.—Stir up 2 to 3 g. of potato starch or 5 g. soluble starch with 100 cc. of 1-per-cent salicylic acid solution, add 300 to 400 cc. boiling water, and boil the mixture until the starch is practically dissolved. Dilute to 1 liter.

Potassium Iodide Solution.—Dissolve 150 g. of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

Hanus Solution.—Dissolve 13.2 g. of iodine in 1000 cc. of glacial acetic acid (99.5-per-cent) that will not reduce chromic acid. Add enough bromine to double the halogen content, determined by titration (3 cc. of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

Standard Sodium Hydroxide Solution.—Prepare a stock concentrated solution of sodium hydroxide by dissolving NaOH in water in the proportion of 200 g. NaOH to 200 cc. water. Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the precipitate of sodium carbonate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off about 175 cc. and dilute to 10 liters with freshly boiled distilled water. Preserve in a stock bottle provided with a large guard tube filled with soda lime. Determine the exact strength by titrating against pure benzoic acid (C_6H_5COOH) using phenolphthalein as indicator. (See Bureau of Standards *Scientific Paper No. 183.*) This solution will be approximately N/4, but do not attempt to adjust it to any exact value. Determine its exact strength and make proper corrections in using it.

Alcoholic Sodium Hydroxide Solution.—Dissolve pure NaOH in 95-per-cent ethyl alcohol in the proportion of about 22 g. per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle, and keep well stoppered. This solution should be colorless or only slightly yellow when used; it will keep colorless longer if the alcohol is previously treated with NaOH (about 80 g. to 1000 cc.), kept at

about 50° C. for 15 days, and then distilled. For an alternate method see *Journal*, American Chemical Society, 1906, p. 395.

Half Normal Sulfuric Acid Solution.—Add about 15 cc. H_2SO_4 (sp. gr. 1.84) to distilled water, cool and dilute to 1000 cc. Determine the exact strength by titrating against freshly standardized NaOH or by any other accurate method. Either adjust to exactly N/2 strength or leave as originally made, applying appropriate correction.

METHODS.

Methods of Testing.

3. The oil shall be tested in accordance with the following methods:

General.—The laboratory sample shall be thoroughly mixed by shaking, stirring, or pouring from one vessel to another and the samples for the individual tests taken from this thoroughly mixed sample.

Loss on Heating at 105 to 110° C.—Place 10 g. of the oil in an accurately weighed 200-cc. Erlenmeyer flask; weigh. Heat in an oven at a temperature between 105 and 110° C. for 30 minutes; cool and weigh. Calculate the percentage loss. This determination shall be made in a current of dry carbon dioxide gas.

Foots.—With all materials at a temperature between 20 and 27° C., mix, by shaking in a stoppered flask for exactly one minute, 25 cc. of the well-shaken sample of oil, 25 cc. of acetone and 10 cc. of the acid calcium chloride solution. Transfer the mixture to a burette where settling can take place for 24 hours. The temperature during this period should be between 20 and 27° C.

The volume of the stratum lying between the clear calcium chloride solution and the clear acetone and oil mixture is read in tenths of a cubic centimeter or a fraction thereof. This reading multiplied by four expresses the amount of foots present as percentage by volume of the oil taken.

Specific Gravity.—Use a pyknometer accurately standardized and having a capacity of at least 25 cc., or any other equally accurate method, making the test at 15.5° C., water being 1 at 15.5° C.

Acid Number.—Weigh from 5 to 10 g. of the oil. Transfer to a 300-cc. Erlenmeyer flask. Add 50 cc. of neutral 95-per-cent ethyl alcohol. Put a condenser loop inside the neck of the flask. Heat on a steam bath for 30 minutes. Cool and add phenolphthalein indicator. Titrate to a faint permanent pink color with the standard sodium hydroxide solution. Calculate the acid number (milligrams KOH per gram of oil).

Saponification Number.—Weigh about 2 g. of the oil in a 300-cc. Erlenmeyer flask. Add 25 cc. alcoholic sodium hydroxide solution.

Put a condenser loop inside the neck of the flask and heat on the steam bath for one hour. Cool, add phenolphthalein as indicator, and titrate with N/2 H_2SO_4. Run two blanks with the alcoholic sodium hydroxide solution. These should check within 0.1 cc. N/2 H_2SO_4. From the difference between the number of cubic centimeters of N/2 H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams KOH required for 1 g. of the oil).

Unsaponifiable Matter.—Weigh 8 to 10 g. of the oil. Transfer to a 250-cc. long-neck flask. Add 5 cc. of strong solution of sodium hydroxide (equal weights of NaOH and H_2O), and 50 cc. 95-per-cent ethyl alcohol. Put a condenser loop inside the neck of the flask and boil for two hours. Occasionally agitate the flask to break up the liquid but do not project the liquid onto the sides of the flask. At the end of two hours remove the condenser and allow the liquid to boil down to about 25 cc.

Transfer to a 500-cc. glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 cc., add 100 cc. redistilled ether. Stopper and shake for one minute. Let stand until the two layers separate sharp and clear. Draw all but one or two drops of the aqueous layer into a second 500-cc. separatory funnel and repeat the process using 60 cc. of ether. After thorough separation draw off the aqueous solution into a 400-cc. beaker, then the ether solution into the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the second separatory funnel and shake out again with 60 cc. of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

Shake the combined ether solution with the accumulated water rinsings and let the layers separate sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 cc. each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stopcock, and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw the ether solution (portionwise if necessary) into a 250-cc. flask and distill off. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool and weigh.

NOTE.—The unsaponifiable oil from adulterated drying oils is volatile and will evaporate on long heating. Therefore heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.

Hanus Iodine Number.—Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer by dropping about 0.15 g. (0.10 to 0.20 g.) to a 500-cc. bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. Add 10 cc. of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc. of chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 cc. of the Hanus solution and let stand with occasional shaking for one-half hour. Add 10 cc. of the 15-per-cent potassium iodide solution and 100 cc. of water, and titrate with standard sodium thiosulfate using starch as indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titration and the titration on the samples and the iodine value of the thiosulfate solution, calculate the iodine number of the samples tested. (Iodine number is centigrams of iodine to 1 g. of sample.)

Color.—Prepare a fresh solution of pure potassium bichromate in pure colorless H_2SO_4 (sp. gr. 1.84). This solution should be in the proportion of 1.0 g. potassium bichromate to 100 cc. (184.0 g.) H_2SO_4. Place the oil and colored solution in separate thin-walled, clear glass tubes of the same diameter (1 to 2 cm.) to a depth of not less than 2.5 cm. and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

TENTATIVE SPECIFICATIONS
FOR
FOOTS PERMISSIBLE IN PROPERLY CLARIFIED PURE RAW LINSEED OIL FROM NORTH AMERICAN SEED[1]

Serial Designation: D 51 – 18 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1918.

I. PERCENTAGE OF FOOTS.

Percentage of Foots. 1. The amount of foots in properly clarified pure raw linseed oil from North American seed, as determined by the test specified below, shall not exceed 2 per cent by volume.

II. METHOD OF DETERMINATION.

Reagents Required. 2. The following reagents are required:

(*a*) Acetone that will pass United States Pharmacopœia specifications.

(*b*) Acid $CaCl_2$ solution, made by saturating with $CaCl_2$ a mixture of 90 parts water and 10 parts concentrated HCl, sp. gr. 1.2, at room temperature.

Operation. 3. With all materials at a temperature between 70 and 80° F., mix by shaking in a stoppered flask for exactly one minute, 25 cc. of the well-shaken sample of oil, 25 cc. of acetone and 10 cc. of the acid $CaCl_2$ solution. Transfer the mixture to a burrette where settling can take place for 24 hours. The temperature during this period should be between 70 and 80° F.

The volume of the strata lying between the clear calcium-chloride solution and the clear acetone and oil mixture is read in tenths of a cubic centimeter or a fraction thereof. This reading multiplied by four expresses the amount of foots present as percentage by volume of the oil taken.

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

TENTATIVE SPECIFICATIONS

FOR

PURITY OF RAW LINSEED OIL FROM SOUTH AMERICAN SEED.[1]

Serial Designation: D 77 – 21 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921.

I. PROPERTIES AND TESTS.

Properties. 1. Properly clarified raw linseed oil from South American seed shall conform to the following requirements:

	Maximum.	Minimum.
Specific Gravity at 15.5°/15.5° C.	0.9360	0.9310
Acid Number	6.00	......
Saponification Number	195	189
Unsaponifiable Matter, per cent	1.50	......
Refractive Index at 25° C.	1.4805	1.4780
Iodine Number (Hanus)	......	170

II. METHODS OF TESTING.

Methods of Testing. 2. The oil shall be tested in accordance with the methods recommended in Section 2 of the Standard Specifications for Purity of Raw Linseed Oil from North American Seed (Serial Designation: D 1) of the American Society for Testing Materials.[2]

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

[2] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

PURITY OF BOILED LINSEED OIL FROM SOUTH AMERICAN SEED.[1]

Serial Designation: D 78 – 21 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921.

I. PROPERTIES AND TESTS.

1. Boiled linseed oil from South American seed shall conform to the following requirements: **Properties.**

	Maximum	Minimum
Specific Gravity at 15.5°/15.5° C	0.945	0.936
Acid Number	8.00	......
Saponification Number	195	189
Unsaponifiable Matter, per cent	1.50	......
Refractive Index at 25° C	1.4840	1.4780
Iodine Number (Hanus)	......	168
Ash, per cent	0.7	0.2
Manganese, per cent	......	0.03
Calcium, per cent	0.3	......
Lead, per cent	......	0.1

II. METHODS OF TESTING.

2. The oil shall be tested in accordance with the methods recommended in Section 2 of the Standard Specifications for Purity of Boiled Linseed Oil from North American Seed (Serial Designation: D 11) of the American Society for Testing Materials.[2] **Methods of Testing.**

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

[2] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

DRY BLEACHED SHELLAC[1]

Serial Designation: D 207 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

I. GRADES

Grades.

1. Dry bleached shellac[2] shall be furnished in two grades, namely, regular and refined.[3]

II. PROPERTIES AND TESTS

Properties.

2. Dry bleached shellac shall conform to the following requirements:

	REGULAR	REFINED
Iodine number, maximum	10	10
Matter insoluble in hot alcohol, maximum	1.0 per cent	0.2 per cent
Moisture, maximum	5.0 "	5.0 "
Wax, maximum	5.5 "	0.2 "
Matter soluble in water, maximum	1.0 "	0.3 "
Ash, maximum	1.0 "	0.3 "

III. METHODS OF TESTING

3. The determination of iodine number, matter insoluble in hot alcohol and moisture shall be made in accordance with the Standard Methods of Testing Shellac (Serial Designation: D 29) of the American Society for Testing Materials.[4]

4. The determination of wax shall be made in accordance with the Standard Method of Test for Determination of Wax in Shellac

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

[2] This material is white shellac. It is also known as "bonedry," "vacdry" or "kilndried" bleached shellac.

[3] Refined bleached shellac has been treated in the process of manufacture so as to remove practically all wax and matter insoluble in hot alcohol.

[4] 1924 Book of A.S.T.M. Standards.

(Serial Designation: D 29) of the American Society for Testing Materials.[1]

5. The determination of matter soluble in water shall be made as follows:

Ten to twenty-five grams of dry shellac shall be ground so that 100 per cent shall pass through a No. 20 sieve (840 micron opening). The shellac shall be placed in a 250-cc. beaker or flask and 100 cc. of distilled water added. It shall be stirred thoroughly, covered with a watch glass and permitted to stand at room temperature (21° C.) for four hours, with occasional stirring.

It shall be decanted through a 12.5-cm. filter paper into a weighed evaporating dish, washing the shellac and filter with at least 50 cc. more of water. It shall be evaporated and the extract dried at 105° C. for one hour or more to constant weight. The increase in weight shall be reported as the water-soluble extract.

6. The determination of ash shall be made as follows:

Approximately 2 to 3 g. of the sample shall be accurately weighed, transferred to a weighed porcelain or platinum crucible and ignited at as low a temperature as possible until all organic matter has been destroyed. The crucible and contents shall be cooled and weighed and the percentage of ash calculated.

[1] A.S.T.M. Standards Adopted in 1925.

TENTATIVE SPECIFICATIONS

FOR

IRON OXIDE AND IRON HYDROXIDE[1]

Serial Designation: D 84 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1924, 1925.

Scope.

1. These specifications cover iron oxide and iron hydroxide pigments of red and brown colors.

I. MANUFACTURE.

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be very finely ground iron oxide or iron hydroxide or a mixture thereof. Siliceous minerals may be present and, if necessary, sufficient carbon pigment may be added to produce the desired color.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

II. PROPERTIES AND TESTS.

Composition and Properties.

3. (*a*) *Dry Pigment.*—The pigment shall conform to the following requirements:

	MAXIMUM.	MINIMUM.
Ferric oxide, per cent	...	30.0
Material other than ferric oxide, insoluble siliceous matter and loss on ignition, per cent	10.0	...
Coarse particles retained on a Standard No. 325 screen,[2] per cent	3.0	...

Organic coloring matters shall be absent.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

[2] For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.) the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

The color and color strength, when specified, and the drying time with a vehicle which has been mutually agreed on, shall be equal to those of an agreed sample.

(*b*) *Paste.*—The paste as received and three months thereafter shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily with linseed oil, turpentine or volatile mineral spirits, or any combination of these, without curdling.

The paste shall conform to the following requirements:

	Maximum.	Minimum.
Pigment, per cent	72.0	68.0
Linseed oil, per cent	32.0	28.0
Moisture and other volatile matter, per cent	0.7	....
Coarse particles and skins (total residue retained on a Standard No. 325 screen,[1] based on pigment), per cent	3.5	....

Number of Tests

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

[1] For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS
FOR
LITHOPONE[1]

Serial Designation: D 208 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925.

Scope.

1. These specifications cover the pigment consisting of zinc sulfide and barium sulfate, commercially known as lithopone. The pigment may be purchased in the dry form or ground in oil to form a paste.

I. MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be made by suitable treatment of a mixture of precipitated zinc sulfide and precipitated barium sulfate.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The behavior on exposure to light, the mixing properties with an approved vehicle, the final consistency with this vehicle and the color shall be equal to, and the brightness and tinting strength (or hiding power) shall be not less than, those of a sample, mutually agreed on by buyer and seller.

(*b*) *Dry Pigment.*—The dry pigment shall meet the following requirements:

	Maximum	Minimum
Coarse particles and skins (total residue retained on a standard No. 325 screen), per cent	1.0	....
Zinc sulfide, per cent	...	26.0
Zinc oxide, per cent	1.0	....
Material soluble in water, per cent	0.8	....
Barium sulfate	The remainder	

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

	Maximum	Minimum
Pigment, per cent	80.0	76.0
Linseed oil, per cent	24.0	20.0
Coarse particles and skins (total residue retained on a standard No. 325 screen, based on pigment), per cent	1.5	....

Number of Tests.

4. (*a*) *Paste.*—One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

(*b*) *Dry Pigment.*—Dry pigment shall be sampled by taking a portion from every tenth barrel or from every one hundredth bag of the dry pigment.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS

FOR

LAMPBLACK[1]

Serial Designation: D 209 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

I. MANUFACTURE

Scope.

1. These specifications cover the pigment commonly known as lampblack. The pigment may be purchased in the dry form, or ground in oil or in japan to form a paste.

Process.

2. (*a*) The dry pigment shall be made by burning oils or tars in such a manner as to form a deposit of carbon or soot. The pigment shall be high grade in every respect, shall be free from oil, greasy matter and from admixture of any other substance.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than that of a sample mutually agreed on by buyer and seller.

(*b*) *Dry Pigment.*—The dry pigment shall meet the following requirements:

Coarse particles retained on a standard No. 325 screen, maximum, per cent	1.0
Ash, maximum, per cent	1.0
Benzol extract (which must be colorless) maximum, per cent	0.5
Tone when diluted with zinc oxide	Clear-blue-gray

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

	MAXIMUM	MINIMUM
Pigment, per cent	40	25
Linseed oil, per cent	75	60
Material volatile at 105° C	0.7	..
Coarse particles and skins left on a No. 325 screen (per cent of the dry pigment)	1.0	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat. It shall meet the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	40	25
Vehicle, per cent	75	60
Coarse particles and skins retained on a standard No. 325 screen (per cent of the dry pigment)	1.0	..
Non-volatile matter in the vehicle, per cent of the vehicle	..	40

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload. **Number of Tests.**

NOTE.—For determining coarse particles, screens 3 in. in diameter are recom mended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.) the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS

FOR

BONE BLACK[1]

Serial Designation: D 210 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925.

Scope.

1. These specifications cover the pigment commonly known as bone black, ivory black or drop black. It may be purchased in the dry form, or ground in oil or in japan to form a paste.

I. MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The dry pigment shall be made by the calcination of bones and shall be unmixed with any other substance.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment in pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the pigment in high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than, that of a sample mutually agreed on by buyer and seller.

(*b*) *Dry Pigment.*—The dry pigment shall meet the following requirements:

Coarse particles retained on a standard No. 325 screen, maximum, per cent	2.0
Ash, maximum, per cent	85.0
Ash insoluble in acids, maximum, per cent	1.0
Benzol extract (which must be colorless), maximum, per cent	0.5
Tone when diluted with zinc oxide	Clear-blue-gray

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

	MAXIMUM	MINIMUM
Pigment, per cent	55	45
Linseed oil, per cent	55	45
Coarse particles and skins (total residue retained on a standard No. 325 screen, based on pigment), per cent	2.5	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat. It shall meet the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	55	45
Vehicle (japan), per cent	55	45
Coarse particles and skins (total residue retained on a standard No. 325 screen, based on pigment), per cent	2.5	..
Non-volatile matter in the vehicle, per cent of the vehicle	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS
FOR
CHROME YELLOW[1]

Serial Designation: D 211 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

Scope. 1. These specifications cover the pigments commonly known as lemon chrome yellow, medium chrome yellow, and orange chrome yellow. The pigment may be purchased in the dry form or ground in oil, or in japan, to form a paste.

I. MANUFACTURE

Process. 2. (*a*) *Dry Pigments.*—The dry pigments, lemon chrome yellow and orange chrome yellow, shall be chemical precipitates consisting of normal or basic lead chromates or mixtures of these with or without admixtures of other insoluble compounds of lead, but without any other admixtures.

Dry medium chrome yellow shall be chemically precipitated normal lead chromate.

(*b*) *Pastes in Oil.*—The pastes in oil shall be made by thoroughly grinding the specified pigments with pure raw or refined linseed oil.

(*c*) *Pastes in Japan.*—The pastes in japan shall be made by thoroughly grinding the specified pigments in high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties. 3. (*a*) The color and tone shall be equal to, and the tinting strength not less than, that of a sample mutually agreed on by buyer and seller.

(*b*) The dry pigments shall meet the following requirements:

	LEMON CHROME ORANGE CHROME	MEDIUM CHROME
Total matter soluble in water, maximum, per cent	0.5	0.5
Total of all substances other than insoluble compounds of lead, maximum, per cent	3.0	3.0
Organic colors or lakes	None	None

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

	Lemon Chrome Orange Chrome	Medium Chrome
Normal lead chromate ($PbCrO_4$), minimum, per cent	..	97.0
Coarse particles (total residue on a No. 325 screen), per cent, not over	1.0	1.0

(c) *Paste in Oil.*—The pastes as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. They shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances.

The pastes shall meet the following requirements:

	Lemon Chrome Orange Chrome		Medium Chrome	
	Maximum	Minimum	Maximum	Minimum
Pigment, per cent	85	65	82	75
Linseed oil, per cent	35	15	25	18
Water and other volatile matter, per cent	0.7	..	0.7	..
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	1.5	..	1.5	..

(d) *Paste in Japan.*—The pastes as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard flat coat. They shall meet the following requirements:

	Lemon Chrome Orange Chrome		Medium Chrome	
	Maximum	Minimum	Maximum	Minimum
Pigment, per cent	85	65	82	75
Vehicle (japan) per cent	35	15	25	18
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	1.5	..	1.5	..
Non-volatile matter in the vehicle, per cent of the vehicle	..	40	..	40

Number of Tests

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS

FOR

PURE CHROME GREEN[1]

Serial Designation: D 212 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

Scope.

1. These specifications cover what is commercially known as pure chrome green. The pigment may be purchased in the dry form or ground in oil or in japan to form a paste.

I. MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be a co-precipitated mixture of lead chromate and iron ferro and ferricyanide blue, with or without other insoluble compounds of lead.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment with high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than, that of a sample mutually agreed on by buyer and seller.

(*b*) *Dry Pigment.*—The dry pigment shall meet the following requirements:

Percentage of total lead present in the form of chromate........	75
Total impurities, maximum, per cent...........................	3
Coarse particles (total residue retained on a No. 325 screen), maximum, per cent..	1.0

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

	Maximum	Minimum
Pigment, per cent	75	65
Linseed oil, per cent	35	25
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	1.5	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat. The paste shall meet the following requirements:

	Maximum	Minimum
Pigment, per cent	75	65
Vehicle (japan), per cent	35	25
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	1.5	..
Non-volatile matter in vehicle, per cent of the vehicle	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE SPECIFICATIONS

FOR

REDUCED CHROME GREEN[1]

Serial Designation: D 213 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

Scope.

1. These specifications cover what is known commercially as reduced chrome green, also known as grinders green. The pigment may be purchased in the dry form or ground in oil or in japan to form a paste.

I. MANUFACTURE

Process.

2. (*a*) *Dry Pigment.*—The pigment shall be a mixture of lead chromate and iron ferro and ferricyanide blue, with or without other insoluble compounds of lead, co-precipitated on a base of barium sulfate or insoluble siliceous material or any mixture thereof.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan.

II. PROPERTIES AND TESTS

Composition and Properties.

3. (*a*) The color and tone shall be equal to, and the tinting strength not less than, that of a sample mutually agreed on by buyer and seller.

(*b*) The dry pigment shall meet the following requirements:

	Maximum	Minimum
Sum of the barium sulfate and insoluble siliceous material, per cent	80	..
Color (total of insoluble lead compounds and iron blue), per cent	..	20
Percentage of total lead present in the form of chromate	..	75
Total calcium oxide in any form soluble in acid, per cent	0.5	..
Coarse particles (total residue retained on a No. 325 screen), per cent	1.5	..

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

(*c*) *Paste in Oil.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances. The paste shall meet the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	88	80
Linseed oil, per cent	20	12
Coarse particles and skins (total residue retained on a No. 325 screen), per cent of the pigment	2.0	..

(*d*) *Paste in Japan.*—The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard flat coat. It shall meet the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	88	80
Vehicle (japan), per cent	20	12
Coarse particles and skins (total residue retained on a No. 325 screen), per cent	2.0	..
Non-volatile matter in vehicle, per cent of the vehicle	..	40

Number of Tests.

4. One sample shall be taken at random from each lot of 1000 packages or less. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

NOTE.—For determining coarse particles, screens 3 in. in diameter are recommended. The screen cloth is described as follows: No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent.

TENTATIVE METHODS OF TESTING SHELLAC VARNISH[1]

Serial Designation: D 214 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting **criticism** and suggestions. It is not a Standard of the Society and is subject to annual **revision.**

Issued, 1925

Shellac varnishes are made by dissolving shellac in alcohol and are designated in the trade in terms of pounds of shellac per gallon of alcohol, the 4, 4.5 and 5-lb. cut goods being most commonly used. Specially denatured alcohol, Formula No. 1 is largely used, and occasionally wood alcohol and butyl alcohol. Pure grain alcohol, or Formula No. 35 denatured is used for confectioner's varnish or glaze.

DETERMINATION OF BODY (SOLIDS)

Apparatus

1. The apparatus shall consist of the following:

(*a*) A weighing bottle for volatilized liquids or a Grethan pipette.

(*b*) A tin-foil dish, $1\frac{1}{4}$ in. in height and $2\frac{1}{2}$ in. in diameter.

(*c*) Prepared Sand, prepared as follows: Take sea-sand, sieve it to take out all foreign material. Digest with hot HCl for about one hour. Wash with water to remove all the acid and soluble impurities. Ignite and put in a desiccator to cool. Then put it in a bottle and stopper it tightly.

Procedure

Procedure. 2. (*a*) Put 10 g. of the prepared sand and a short glass rod into the tin-foil dish. Weigh them all together and record the weight accurately. Run into the dish about 1 g. of the sample from the weighing bottle. Record the exact weight of the sample taken into the dish.

(*b*) Mix the varnish and sand with the glass rod.

Note.—This is especially important in testing varnish of over 6 lb. so that the varnish and sand will be intimately mixed.

Place in an oven at a temperature of 105° C. and heat for one hour. At the end of one hour take out, cool and weigh. The increase

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

in weight of the tin-foil, sand and glass rod is the weight of the shellac in the varnish.

3. (*a*) In the case of orange shellac varnish, divide the percentage of the shellac found by 0.98 because orange shellac loses 2 per cent in heating for an hour at 105° C. **Calculation.**

(*b*) In the case of white shellac varnish, divide the percentage of shellac found by 0.95 because white shellac varnish is made from bone dry shellac, which contains 5 per cent of moisture.

Example.—

Orange Shellac Varnish:

Weight of varnish taken.................................. 1.3200 g.
Weight of residue (after drying)......................... 0.5491 g.

$$\text{Per cent of body} = \frac{0.5491}{0.98 \times 1.3200} \times 100 = 42.45 \text{ per cent}$$

This percentage corresponds to 5 lb. per gallon.

White Shellac Varnish:

Weight of varnish taken.................................. 1.2973 g.
Weight of residue (after drying)......................... 0.5235 g.

$$\text{Per cent of body} = \frac{0.5235}{0.95 \times 1.2973} \times 100 = 42.45 \text{ per cent}$$

This percentage corresponds to 5 lb. per gallon.

(*c*) To convert the percentage of solids (residue) of the varnish into pounds per gallon, from the percentage of solids expressed in pounds of solids in 100 lb. of varnish, the pounds of solvent is found by difference. The weight of solvent divided by 6.7793 (the weight of one gallon of denatured alcohol (Formula No. 1) at 16° C. (60° F.) gives the number of gallons of alcohol present. From this the number of pounds of shellac per gallon of alcohol is determined.

Example.—

$100 - 30.68$ (Solids found) $= 69.32$ lb. of alcohol

$$\frac{69.32}{6.7793} = 10.23 \text{ gallons of alcohol.}$$

$$\frac{30.68}{10.23} = 3 \text{ lb. per gallon.}$$

For convenience, tables are appended hereto giving pounds of shellac per gallon for varnishes for various specific gravities.

DETERMINATION OF WAX

4. The wax shall be determined in accordance with the Standard Method of Test for Determination of Wax in Shellac (Serial Designation: D 29) of the American Society for Testing Materials.[1]

[1] A.S.T.M. Standards Adopted in 1925.

DETERMINATION OF PURITY

5. Purity of varnish of both white and orange shellac variety may be ascertained by determining the iodine number on the gum residue. Two methods are available:

Method 1.—Run 0.5 to 0.7 cc. of varnish into a weighed 6 or 8-oz. glass-stoppered Erlenmeyer flask (a light weight bottle with hollow stopper). Add 1 or 2 cc. of alcohol to spread the varnish film from the bottom of the flask. Place the flask in a hot water bath (90° C.) for 30 minutes and aspirate the alcohol vapor. Cool, weigh and add 20 cc. of acetic acid (melting point 14.8° C.). Proceed in accordance with the method for iodine number described in the Determination of Rosin in the Standard Methods of Testing Shellac (Serial Designation D 29) of the American Society for Testing Materials.[1]

Method 2.—

APPARATUS

A weighing bottle for the adulteration of liquids, or a Grethan pipette.

PROCEDURE

With the percentage of solids known, weigh out enough of the original varnish to correspond to 0.200 g. of solids (0.450 to 0.500 g. of varnish). This is satisfactory in the case of slight adulteration. If heavy adulteration is suspected, weigh out an amount of varnish to correspond to 0.150 g. of solids or less.

From the weighing bottle or pipette weigh, by difference, the amount of varnish selected into a 250-cc. glass-stoppered bottle. Warm in a water bath at not over 78° C. for about 15 minutes and aspirate the alcohol vapor until practically dry. Cool, weigh and add 20 cc. of acetic acid (melting point 14.8° C.). Proceed in accordance with the method for iodine number described in the Determination of Rosin in the Standard Methods of Testing Shellac (Serial Designation: D 29) of the American Society for Testing Materials.[1]

6. *Qualitative Test.*—A qualitative test may be applied to the varnish, and if this shows only adulteration with copal the amount of copal may be approximately determined from the iodine number with the iodine number of copal taken as 130 for purposes of calculation.

In making the qualitative test, filter some of the varnish through dry paper into a large test tube (6 by $\frac{3}{4}$ in.). When 10 cc. has run through, fill the test tube with 99-per-cent methyl alcohol and agitate.

[1] 1924 Book of A.S.T.M. Standards.

A pure shellac will remain clear and when adultered with copal will give a precipitation on standing.

A qualitative test for rosin may be made by the Halphen-Hicks method as described by Walker and Steele, *Bulletin No. 232*, Bureau of Standards, March, 1923, pp. 284–285.

COMPLETE VARNISH ANALYSIS

7. A complete varnish analysis would include distilling about 50 cc. of varnish to dryness on a sand bath and redistilling the distillate to determine the boiling point. A portion of the final distillate should be diluted with water to ascertain if other solvents, such as naphtha or benzol, have been added. These will separate out and the separated layer may be measured.

APPENDIX

RELATION OF POUNDS OF SHELLAC PER GALLON TO SPECIFIC GRAVITY

WHITE SHELLAC VARNISH: DRY BLEACHED SHELLAC (5 PER CENT MOISTURE), DENATURED ALCOHOL, FORMULA NO 1, 190 PER CENT PROOF

Shellac, lb. per gal. of Alcohol	Specific Gravity, 16° C. (60° F.)	Deg. Baumé	Weight per 1 Gal. Varnish, lb.
3	0.9056	24.60	7.55
3.5	0.9167	22.70	7.64
4	0.9278	20.90	7.74
4.5	0.9375	19.33	7.79
5	0.9464	17.92	7.89
5.5	0.9530	16.90	7.95
6	0.9597	15.90	8.00

ORANGE SHELLAC VARNISH: TN SHELLAC (2 PER CENT MOISTURE), DENATURED ALCOHOL, FORMULA NO. 1, 190 PER CENT PROOF

Shellac, lb. per gal. of Alcohol	Specific Gravity, 16° C. (60° F.)	Deg. Baumé	Weight per 1 Gal. Varnish, lb.
3	0.900	25.55	7.50
3.5	0.9114	23.6	7.60
4	0.9228	21.7	7.69
4.5	0.9318	20.25	7.77
5	0.9395	19.0	7.83
5.5	0.9500	17.36	7.92
6	0.9554	16.53	7.97

NOTE 1.—Shellac varnishes will not always agree exactly with the above specific gravity, estimated at 16° C. (60° F.). They will vary somewhat, due to more or less moisture and insoluble matter contained in the white and orange shellacs, and also to loss of solvent by evaporation. However, the above figures agree closely with theoretical and practical results.

NOTE 2.—Ten pounds of shellac cut in alcohol yields a volume of 1 gallon. This must be taken into consideration in determining the weight per gallon of varnish.

TENTATIVE METHOD OF TEST

FOR

COARSE PARTICLES IN PAINT PIGMENTS[1]

Serial Designation: D 185 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924

I. APPARATUS

1. The apparatus shall consist of a standard No. 325 sieve.[2] A standard No. 325 sieve for comparison purposes should be reserved in the laboratory. Whenever a new sieve is secured, a practical test of its accuracy should be made by running on it and on the standard sieve a pigment that has a considerable percentage of coarse particles. A reserve stock of such pigment should be kept for this purpose.

II. PROCEDURE

2. The sieve shall be weighed on an analytical balance, the weight being recorded in a figure carried to the third decimal place. The sieve shall then be wet on both sides with the liquid to be used for wash purposes.

3. The sample of the pigment to be tested shall be weighed. For most pigments 10 g. will be the proper quantity. For black pigments of low specific gravity 2 g. will be sufficient. For pigments like Prussian blue and graphite, 3 g. are generally used. The weighed samples of pigment shall then be transferred to the sieve.

If the pigment is one that is difficult to wet with the wash liquid, the quantity weighed out should first be placed in a beaker containing

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

[2] Sieves 3 in. in diameter will be received by the U. S. Bureau of Standards for test. The cloth will be tested to determine whether or not it conforms to the specifications for cloth of the U. S. Standard Sieve Series. The specifications for the sieve frame are now being prepared. No. 325 cloth of the U. S. Standard Sieve Series should be made of wire 0.036 mm. (0.0014 in.) in diameter, a tolerance of 15 per cent under and 35 per cent over being allowed on this diameter. The average opening between adjacent parallel wires should be 0.044 mm. (0.0017 in.), the tolerance being 8 per cent with the additional limitation that the maximum opening shall not exceed 0.044 mm. by more than 90 per cent. Sieves whose cloth conforms to these specifications and whose frames are in accordance with specifications now in preparation will be marked with the letters "BS" and the year in which the test is made. A report will be issued for each sieve submitted, a nominal fee being charged for this test.

some of a liquid that easily wets the pigment and that is miscible with the wash liquid. For example, if water is to be used as the wash liquid, alcohol may be used as the wetting medium. The pigment is then gently stirred with the wetting liquid and the contents of the beaker transferred to the sieve. Where small particles of pigment are retained on the stirring rod or walls of the beaker they may easily be removed with the brush.

4. The sieve shall then be held under a tap delivering about 300 to 500 cc. of wash liquid per minute. By slightly shaking the sieve, the pigment will be rapidly carried through. A soft camel's hair brush may be used in aiding the operation. If the sieve is held at a slight angle so that the pigment will gradually collect at one edge during the washing process, and then rotated, the pigment may be brushed out rapidly, with no risk of clogging the screen.

5. After the majority of the finely divided portion of the pigment has passed through the sieve (from 2 minutes to 1 hour, according to kind of pigment), the sieve shall be placed in an 8-in. porcelain dish containing 250 cc. of the wash liquid. The sieve will thus be covered to a depth of about $\frac{1}{2}$ in. The pigment remaining on the sieve shall be brushed with a soft 1-in. camel's hair brush at the rate of two strokes per second during two periods of 10 seconds each. The sieve shall then be raised from the dish after each 10-second period to let the liquid on the sieve run through. The liquid in the dish should be changed after every two brushing periods described above. This operation shall be continued until typewritten letters can be read through a layer of the wash liquid 8 cm. thick, which is approximately the height of a filled 250-cc. beaker of the low-form (Griffin) type.

Occasionally pigments will be found that foam when water is used as the wash liquid. In such instances, during the last washing in the porcelain dish, the use of a liquid that breaks down the foaming and is readily miscible with water, such as alcohol, will usually overcome this difficulty.

6. The pigment particles adhering to the brush shall then be washed back onto the sieve and the water below the sieve wiped off. A few drops of alcohol and then of ether should be added to expedite drying. The screen shall be dried for one hour in an oven at 105° C., or on a radiator, cooled, weighed, and the percentage of coarse particles calculated.

7. For pigments requiring wash liquids such as turpentine, mineral spirits, or kerosene, the wash liquid shall be siphoned from a vessel onto the sieve, finishing with ether to remove the wash liquid adhering to the sieve.

TENTATIVE METHODS OF ROUTINE ANALYSIS OF WHITE LINSEED OIL PAINTS[1]

Serial Designation: D 215 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

PRELIMINARY PROCEDURE

On receipt of a sample make a record of the label, noting especially the brand, the name of the manufacturer, and any statement as to composition and net contents. Weigh the unbroken package, open, note odor and condition of the contents, pour into a clean container, and mix thoroughly by pouring from one container to the other, finally leaving the well-mixed sample in the second container, which shall be tightly closed. The well-mixed sample is used at once for the determinations described under "Methods." The original can and cover may be cleaned with gasoline, wiped dry, and then weighed. This weight subtracted from the original weight will give the net weight of the contents. If desired, the specific gravity of the paint may be determined and the weight per gallon calculated, and the volume of paint and the capacity of the container may be measured.

REAGENTS REQUIRED

Extraction Mixture.—10 volumes ether (ethyl ether);
6 volumes benzol;
4 volumes methyl alcohol;
1 volume acetone.

Aqueous Sodium Hydroxide.—Dissolve 100 g. of NaOH in distilled water and dilute to 300 cc.

Alcoholic Sodium Hydroxide Solution.—Dissolve pure NaOH in 95-per-cent ethyl alcohol in the proportion of about 22 g. per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

bottle and keep well stoppered. This solution should be colorless or only slightly yellow when used, and it will keep colorless longer if the alcohol is previously treated with sodium hydroxide (about 80 g. to 1000 cc.), kept at about 50° C. for 15 days, and then distilled.

Hanus Solution.—Dissolve 13.2 g. of iodine in 1000 cc. of 99.5-per-cent glacial acetic acid which will not reduce chromic acid. Add enough bromine to double the halogen content as determined by titration (3 cc. of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

Standard Sodium Thiosulfate Solution.—Dissolve pure sodium thiosulfate in distilled water (that has been well boiled to free it from carbon dioxide) in the proportion of 24.83 g. crystallized sodium thiosulfate to 1000 cc. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine, pure potassium biiodate, or pure KIO_3 (see Treadwell-Hall, Analytical Chemistry, Vol. 2.) This solution will be approximately 0.1 *N*, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly 0.1 *N*. Preserve in a stock bottle provided with a guard tube filled with soda lime.

Starch Solution.—Stir up 2 to 3 g. of potato starch or 5 g. of soluble starch with 100 cc. of 1-per-cent salicylic acid solution, add 300 to 400 cc. of boiling water, and boil the mixture until the starch is practically dissolved, then dilute to 1 liter.

Potassium Iodide Solution.—Dissolve 150 g. of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

Acid Ammonium Acetate Solution.—Mix 150 cc. of 80-per-cent acetic acid, 100 cc. of water, and 95 cc. of strong ammonia (sp. gr. 0.90).

Ammonium Polysulfide.—Pass H_2S gas into 200 cc. of strong ammonium hydroxide (sp. gr. 0.90) in a bottle immersed in running water or in iced water until the gas is no longer absorbed; then add 200 cc. of strong ammonium hydroxide (sp. gr. 0.90) and dilute with water to 1000 cc. Digest this solution with 25 g. of flowers of sulfur for several hours and filter.

"Lead Acid."—Mix 300 cc. of H_2SO_4 (sp. gr. 1.84) and 1800 cc. of distilled water. Dissolve 1 g. of c. p. lead acetate in 300 cc. of distilled water and add this to the hot solution, stirring meanwhile. Let stand at least 24 hours and siphon through a thick asbestos filter.

Potassium Permanganate Solution.—Dissolve 3.2 g. of pure potassium permanganate in a liter of distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and

standardize as follows: In a 400-cc. beaker dissolve 0.40 to 0.50 g. of Bureau of Standards' sodium oxalate in 250 cc. of hot water (80 to 90° C.) and add 15 cc. of dilute sulfuric acid (1:1). Titrate at once with the potassium permanganate solution, stirring the liquid vigorously and continuously. The permanganate must not be added more rapidly than 10 to 15 cc. per minute, and the last 0.5 to 1 cc. must be added dropwise with particular care to allow each drop to be fully decolorized before the next is introduced. The temperature of the solution should not be below 60° C. by the time the end point is reached. (Too rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient.) The weight of sodium oxalate used multiplied by 0.8334 gives its iron equivalent. The permanganate solution should be kept in a glass stoppered bottle painted black to keep out light.

The iron (Fe) value of the $KMnO_4$ multiplied by 1.076 theoretically equals its antimony (Sb) equivalent. However, for use in determining antimony, the $KMnO_4$ is best standardized as follows: To 0.25 g. of pure metallic antimony in a 500-cc. Pyrex Erlenmeyer flask, add 12 to 15 cc. of concentrated H_2SO_4 and 10 to 12 g. of K_2SO_4; heat until all the antimony is dissolved, cool, dilute to 250 cc. with water, add 20 cc. of concentrated HCl, cool to 10 to 15° C., and titrate with the $KMnO_4$ solution until a faint pink color is obtained. For special work, after digesting, dilute to 100 cc. with water, add 1 to 2 g. of Na_2SO_3, and boil until all the SO_2 is expelled. This is shown when no blue color is obtained with starch-iodate paper (see below); the volume will be reduced about one-half. Dilute to 250 cc. with water, add 20 cc. of HCl (sp. gr. 1.19), and complete as described.

Standard Potassium Ferrocyanide.—Dissolve 22 g. of the pure salt in water and dilute to 1000 cc. To standardize, transfer about 0.2 g. (accurately weighed) of pure metallic zinc or freshly ignited pure zinc oxide to a 400-cc. beaker. Dissolve in 10 cc. of HCl and 20 cc. of water. Drop in a small piece of litmus paper, add ammonium hydroxide until slightly alkaline, then add HCl until just acid, and then 3 cc. of strong HCl. Dilute to about 250 cc. with hot water and heat nearly to boiling. Run in the ferrocyanide solution slowly from a burette with constant stirring until a drop tested on a white porcelain plate with a drop of the uranyl indicator shows a brown tinge after standing one minute. A blank should be run with the same amounts of reagents and water as in the standardization. The amount of ferrocyanide solution required for the blank should be subtracted from the amounts used in standardization and in titration of the sample. The stand-

ardization must be made under the same conditions of temperature, volume, and acidity as obtained when the sample is titrated.

Uranyl Indicator for Zinc Titration.—A 5-per-cent solution of uranyl nitrate in water or a 5-per-cent solution of uranyl acetate in water made slightly acid with acetic acid.

Alkaline Lead Nitrate Solution.—Into 100 cc. of KOH solution (56 g. in 140 cc. of water) pour a saturated solution of lead nitrate (250 g. in 500 cc. of water) until the precipitate ceases to redissolve, stirring constantly while mixing. Let settle, filter through asbestos, and dilute the clear filtrate with an equal volume of water. About 3 volumes of the lead nitrate solution will be required for one of the KOH.

Ammoniacal Cadmium Chloride or Zinc Sulfate Solution.—Dissolve 8 g. of cadmium chloride in 200 cc. of water and add 200 cc. of NH_4OH (sp. gr. 0.90), or, dissolve 200 g. of zinc sulfate in 1080 cc. of water and 920 cc. of NH_4OH (sp. gr. 0.90).

Standard Potassium Iodate Solution.—Dissolve 3.6 g. of KIO_3 and 39 g. of KI in 1000 cc. of water. For general work the theoretical sulfur titer of this solution should be used; for special work, the solution may be standardized against like material, such as a lithopone of known sulfide sulfur content. The theoretical titer is based on standard $Na_2C_2O_4$ and is obtained as follows: To 300 cc. of water in a 600-cc. flask, preferably glass stoppered, add 10 cc. of concentrated HCl (sp. gr. 1.19) and 1 g. of KI. Cool and add 10 cc. of 0.1 *N* $KMnO_4$ solution which has been standardized against $Na_2C_2O_4$. Swirl gently, stopper, and let stand for five minutes. Titrate the liberated iodine with standard $Na_2S_2O_3$ solution until the color fades. Then add 10 cc. of starch solution and continue the titration until the blue color is destroyed. Repeat the titration with the sole difference that 10 cc. of the iodate solution is substituted for the $KMnO_4$ solution. Calculate the normality of the iodate solution.

Starch Indicator for Sulfur Titration.—(1) To 1000 cc. of boiling water, add a cold suspension of 6 g. of starch in 100 cc. of water and boil vigorously for five minutes. Cool the solution, add 6 g. of $ZnCl_2$ dissolved in 50 cc. of cold water, thoroughly mix and set aside for 24 hours. Decant the clear supernatant liquid into a suitable container, add 3 g. of KI, and mix thoroughly. (2, Optional.) Prepare an emulsion of 6 g. of soluble starch in 25 cc. of water, add a solution of 1 g. of NaOH in 10 cc. of water, and stir the solution until it gelatinizes. Dilute to 1000 cc. with water, add 3 g. of KI, and mix thoroughly.

Starch-Iodate Paper.—Impregnate filter paper with a solution obtained by heating 2 g. of starch with 100 cc. of water, and, after solution, adding 0.2 g. of KIO_3 dissolved in 5 cc. of water.

Standard Iodine Solution for SO_2.—Place 15 to 20 g. of pure KI in a liter flask, dissolve in as little water as possible, and then add about 6.4 g. of resublimed iodine. Shake until the iodine is all dissolved, dilute to the mark with water, and mix. This solution is approximately 0.05 *N* and is standardized against 0.05 *N* $Na_2S_2O_3$ to obtain its true normality.

Standard Sodium Thiosulfate Solution for SO_2.—Prepare and standardize as described above, except that 12.42 g. of pure crystallized $Na_2S_2O_3 \cdot 5H_2O$ are used or the 0.1 *N* solution may be diluted with an equal volume of cold CO_2-free water.

METHODS

WATER (NOTE 1)

Mix 100 g. of the paint in a 250-cc. flask with 75 cc. of toluene. Place the flask in an oil bath, connect with condenser, apply heat to the bath, and distil until about 50 cc. of distillate have been collected in a graduate. The temperature in the flask should be then about 105 to 110° C. The number of cubic centimeters of water collected under the toluene in the receiver is the percentage of water in the paint.

VOLATILE THINNER

Weigh accurately from 3 to 5 g. of the paint into a tared flat-bottomed dish about 8 cm. in diameter, spreading the paint over the bottom. Heat at 105 to 110° C. for one hour, cool, and weigh. Calculate the loss in weight as percentage of water and volatile thinner, subtract from this the percentage of water (1), and report the remainder as volatile thinner.

NATURE OF THE THINNER

Transfer about 150 g. of the paint to a 500-cc. flask fitted with a 2-hole cork stopper carrying a spray trap connected with a vertical condenser. Through the other hole in the stopper pass an influx tube for steam. (This tube should dip below the surface of the paint.) Heat the flask in an oil bath or an air bath at 100° C. and pass through it a current of steam; with the steam still passing through, raise the temperature of the bath to 130° C. Catch the distillate in a small separatory funnel; continue distillation until 300 cc. of water has been condensed. Portions of this water may be drawn from the cock of the separatory funnel from time to time, but care must be taken not to draw out any of the volatile thinner. Let the distillate stand until it separates into two layers, then draw off the water, and filter the volatile thinner through a dry filter paper into a dry flask. If the

thinner is apparently turpentine, examine the distillate by the methods described under the Standard Specifications for Turpentine (Serial Designation: D 13) of the American Society for Testing Materials.[1] If the thinner is a mixture of turpentine and mineral spirits, an approximate determination of the amount of turpentine may be made by the polymerization test specified for under turpentine. It should be noted that turpentine is slightly soluble in water (about 0.3 to 0.4 cc. per 100 cc. of water).

To test for benzol, add a few drops of the distillate to a small quantity of a mixture of concentrated HNO_3 and concentrated H_2SO_4, and heat cautiously. The characteristic odor of nitrobenzol will be noted if benzol is present.

If the thinner is apparently all mineral spirits, no further examination is necessary.

Percentage of Pigment

Strain a portion of the well-mixed sample through a No. 80 sieve to remove any skins and weigh accurately about 15 g. of the strained paint in a weighed centrifuge tube. Add 20 to 30 cc. of "extraction mixture" (see "Reagents"), mix thoroughly with a glass rod, wash the rod with more of the extraction mixture, and add enough of the reagent to make a total of 60 cc. in the tube. Place the tube in the container of a centrifuge, surround the tube with water, and counterbalance the container of the opposite arm with a similar tube, or a tube with water. Whirl at a moderate speed until well settled. Decant the clear supernatant liquid, repeat the extraction twice with 40 cc. of extraction mixture and once with 40 cc. of ethyl ether. After drawing off the ether, set the tube in a beaker of water at about 80° C. or on top of a warm oven for 10 minutes, then in an oven at 105 to 110° C. for two hours. Cool, weigh, and calculate the percentage of pigment. Grind the pigment to a fine powder, pass through a No. 80 sieve to remove any skins, and preserve in a stoppered bottle.

Percentage of Non-volatile Vehicle

Add together the percentages of water, of volatile thinner, and of pigment, and subtract the sum from 100. Report the remainder as non-volatile vehicle.

Testing Non-volatile Vehicle

(*a*) *Preparation of fatty acids.*

To about 25 g. of the paint in a porcelain casserole, add 15 cc. of aqueous sodium hydroxide (see "Reagents") and 75 cc. of ethyl

[1] 1924 Book of A.S.T.M. Standards.

alcohol, mix and heat uncovered on a steam bath until all volatile thinner is driven off and saponification is complete. Add 100 cc. of water, boil, add H_2SO_4 (sp. gr. 1.2) (8 to 10 cc. in excess), boil, stir, and transfer to a separatory funnel to which some water has been previously added. Draw off as much as possible of the acid aqueous layer and any insoluble or precipitated matter, wash once with water, then add 50 cc. of water and 50 cc. of ethyl ether. Shake very gently with a whirling action to dissolve the fatty acids in the ether, but not so violently as to form an emulsion. Draw off the aqueous layer and wash the ether layer with one 15-cc. portion of water and then with 5 cc. portions of water until free from sulfuric acid. Then draw off the water layer completely. Transfer the ether solution to a dry flask and add 25 to 50 g. of anhydrous sodium sulfate. Stopper the flask and let stand with occasional shaking at a temperature below 25° C. until the water is completely removed from the ether solution, which will be shown by the solution becoming perfectly clear above the solid sodium sulfate. Decant this clear solution, if necessary, through a dry filter paper into a dry 100-cc. Erlenmeyer flask. Pass a rapid current of dry air (pass through a $CaCl_2$ tower) into the mouth of the Erlenmeyer flask and heat to a temperature below 75° C. on a dry hot plate until the ether is entirely driven off. It is important to follow all of the details, since ether generally contains alcohol, and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solution and subsequent drying with anhydrous sodium sulfate removes both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from fatty acids by gentle heat. If the pigment settles out rapidly in a sample of the paint on standing so that sufficient vehicle can be poured off; or, if sufficient vehicle is obtained by centrifuging the paint, it will be advantageous to saponify this separated vehicle and liberate and prepare the fatty acids as described.

The fatty acids prepared as above should be kept in a stoppered flask and examined at once.

(*b*) *Test for mineral oil and other unsaponifiable matter.*

Place 10 drops of the fatty acids Method (*a*), in a 50-cc. test tube, add 5 cc. of alcoholic soda (see "Reagents"), boil vigorously for five minutes, add 40 cc. of water, and mix; a clear solution indicates that not more than traces of unsaponifiable matter are present.

(*c*) *Iodine number of fatty acids* (Note 2).

Place a small quantity of the fatty acids Method (*a*), in a small weighing burette or beaker. Weigh accurately. Transfer by dropping

about 0.15 g. (0.10 to 0.20 g.) into a 500-cc. bottle having a well ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. (If desired the sample may be weighed in a small wide-mouthed vial and the vial containing the weighed sample placed in the bottle or flask.) Add 10 cc. of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc. of chloroform to two empty bottles like that used for the sample. Add to each bottle 25 cc. of the Hanus solution (see "Reagents") and let stand with occasional shaking for 30 minutes. Add 10 cc. of the 15-per-cent potassium iodide solution and 100 cc. of water, and titrate with standard sodium thiosulfate solution (see "Reagents"), using starch as indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titrations and the titration on the sample and the iodine value of the thiosulfate solution, calculate the iodine number of the sample tested. (Iodine number is centigrams of iodine to 1 g. of sample.)

Analysis of Pigment

(*a*) *Qualitative Analysis.*

A complete qualitative analysis, following the well-established methods, should be made and the quantitative scheme modified as required. Add acetic acid slowly to the pigment until all carbonate is decomposed (noting whether any hydrogen sulfide is evolved); then add a large excess of acid ammonium acetate solution (see "Reagents"), boil, filter, and test the filtrate for metals other than lead and zinc (especially calcium and barium). The absence of calcium in this filtrate indicates that the extending pigments contain no calcium carbonate or calcium sulfate; the absence of barium indicates that the extending pigments contain no barium carbonate (Note 5). Wash the matter insoluble in acid ammonium acetate solution with another portion of this solution, and finally with hot water. This insoluble matter is dried, ignited, and tested for siliceous matter, barium sulfate, and titanium compounds. To test for the latter, place a small amount of the insoluble matter, or of the original sample (about 0.5 g.), in a 250-cc. Pyrex glass beaker; add 20 cc. of concentrated H_2SO_4 and 7 to 8 g. of ammonium sulfate. Mix well, and boil for a few minutes. A residue denotes the presence of silica or siliceous matter. Cool the solution, dilute with 100 cc. of water, heat to boiling, settle, filter, wash with hot 5-per-cent sulfuric acid until free from titanium. The residue may be tested for lead, barium, and silica.

Add hydrogen peroxide to a small portion of the filtrate; a clear yellow-orange color indicates the presence of titanium. Boil another portion of the filtrate with metallic tin or zinc; a pale blue to violet coloration indicates titanium. Treat another portion (about 1 g.) of the pigment with 20 cc. of HCl (1:1) and note whether any H_2S is evolved; boil the solution for about 5 minutes, add about 25 cc. of hot water, filter, and wash with hot water. Render a small portion of the filtrate alkaline with NH_4OH, acidify with HCl, and add a little $BaCl_2$ solution; a white precipitate ($BaSO_4$) indicates the presence of a soluble sulfate. To another portion of the filtrate add a little H_2SO_4; a white precipitate indicates the presence of lead, soluble barium or both (some $CaSO_4$ may also separate); filter, wash to remove free acid, and treat the precipitate with a few drops of KI solution; the formation of yellow PbI_2 indicates the presence of lead. The white precipitate may also be treated with H_2S water; the formation of black PbS indicates the presence of lead. To another portion of the original filtrate add NH_4OH until alkaline, render slightly acid with acetic acid, heat to boiling, and add a little $K_2Cr_2O_7$ solution; a yellow or orange-yellow precipitate indicates the presence of lead, soluble barium or both. To another portion of the original filtrate add a few drops of $K_4Fe(CN)_6$ solution; a white precipitate with a blueish tinge indicates the presence of zinc. Pass into the remaining portion of the original filtrate a current of H_2S for 5 to 10 minutes, add an equal volume of water and pass H_2S into the solution for about 5 minutes; filter, wash with H_2S water; then digest the precipitate with ammonium polysulfide, filter, acidify the filtrate with HCl and warm; the presence of antimony is indicated by the separation of an orange colored precipitate. The filtrate from the H_2S precipitate may be tested for barium, calcium, and magnesium in the usual manner.

(*b*) *Quantitative Analysis.*

(1) *Single Pigments:*

If the sample is a single pigment, follow the method described in the Standard Methods of Routine Analysis of White Pigments (Serial Designation: D 34) of the American Society for Testing Materials[1] for the particular pigment in hand.

(2) *Mixed or Composite Pigments:*

Moisture (Note 3) (*Matter Volatile at 105–110° C.*).—Place 1 to 2 g. of the sample in a wide-mouth, short weighing tube provided with glass stopper. Heat with the stopper removed for 2 hours at a tem-

[1] 1924 Book of A.S.T.M. Standards.

perature between 105 and 110° C. Insert the stopper, cool, and weigh. Calculate the loss in weight as moisture (matter volatile at 105 to 110° C.).

Loss on Ignition.—Ignite 1 g. of the pigment in a porcelain crucible over a Meker burner to constant weight (Note 4).

Insoluble Matter.—Moisten 1 g. of the sample with a few drops of alcohol, cover, add 40 cc. of HCl (1:1), boil gently for 5 to 10 minutes. Wash off cover, evaporate to dryness, and heat at about 150° C. for one-half to one hour to dehydrate the residue. Moisten the residue with 4 cc. of concentrated HCl, allow to stand a few minutes, dilute with 100 cc. hot water, boil a few minutes, filter hot through paper, wash with hot water (till washings give no test for lead and chlorine). Ignite the paper and residue in a platinum or porcelain crucible, cool, and weigh total insoluble matter (Note 5). (The insoluble matter may be filtered off on a Gooch crucible, washed with hot water, dried at 105° C., cooled, and weighed; then ignited, cooled, and weighed, when it is desired to get the loss on ignition (combined water, organic matter, etc.) of same, or the insoluble matter is not to be further examined.) If the sample contains titanium pigment, practically all of the TiO_2 will be found in the insoluble matter along with $BaSO_4$ and siliceous matter. The TiO_2 may be determined in the insoluble matter or in a separate portion of the original sample by the method described in the Standard Methods of Routine Analysis of Titanium Pigments (Serial Designation: D 186) of the American Society for Testing Materials.[1] To determine $BaSO_4$, mix the ignited insoluble matter with about 10 times its weight of anhydrous sodium carbonate (grinding the mixture in an agate mortar if necessary), and fuse the mixture in a covered platinum crucible, heating about one hour. Let cool, place crucible and cover in a 250-cc. beaker, add about 100 cc. of water, and heat until the melt is disintegrated. Filter on paper (leaving crucible and cover in beaker) and wash the beaker and filter thoroughly with hot water to remove soluble sulfates. Place the beaker containing the crucible and cover under the funnel, pierce the filter with a glass rod, and wash the residue into the beaker by means of a jet of hot water. Wash the paper with hot dilute HCl (1:1) and then with hot water. Remove crucible and cover, washing them with a jet of hot water and removing any adhering precipitate. Add cautiously 20 cc. of concentrated H_2SO_4 and evaporate until fumes of H_2SO_4 are evolved and the precipitated matter is dissolved. *Cool*, add cautiously, with stirring, about 100 cc. of water, and boil a few minutes. Let the precipitate

[1] A.S.T.M. Standards Adopted in 1925.

settle, filter on a weighed Gooch crucible, wash with hot water, ignite, cool, and weigh as $BaSO_4$. Subtract the sum of the percentages of $BaSO_4$ and TiO_2 from the percentage of total insoluble matter and report the result as the percentage of insoluble siliceous matter (Note 6).

If it is desired to examine the siliceous matter, unite the filtrates from the Na_2CO_3 fusion and the $BaSO_4$ precipitate, acidify with HCl, evaporate to dryness, and proceed as in a silicate analysis, taking cognizance of any TiO_2 that may be found, if titanium pigment were originally present.

Total Lead (*Antimony*).—Unite the filtrate and washings (total volume 150 to 200 cc.) from the total insoluble matter, pass H_2S into the solution until it is saturated, add an equal volume of water, and again saturate with H_2S. Filter, wash with water containing a little hydrogen sulfide, dissolve in hot HNO_3 (1:3), washing the paper with hot water; add 10 to 20 cc. of H_2SO_4 (1:1), evaporate until copious fumes of sulfuric acid are evolved; cool, add about 75 cc. of water, and then about 75 cc. of 95-per-cent ethyl alcohol. Stir, let settle, filter on a Gooch crucible, wash with dilute alcohol, dry in an oven at 105 to 110° C.; or, ignite gently in a radiator[1] or muffle, cool, and weigh as $PbSO_4$. Calculate to PbO (Note 7).

If the pigment contains antimony, filter and wash the sulfide precipitate as above; then wash the precipitate with a fine jet of H_2O from the paper into a porcelain dish or casserole, add 25 cc. of ammonium polysulfide (see "Reagents"), cover the vessel, and warm the mixture at 40 to 60° C. for 10 to 15 minutes with frequent stirring. Wash off cover, filter through the paper used in the first case, and wash with 2 to 3-per-cent Na_2S or $(NH_4)_2S$ solution. Discard the filtrate. Dissolve the residue in hot dilute HNO_3 (1:3), and determine the lead as $PbSO_4$, as described above. Or, the original sulfide precipitate may be discarded and the lead determined on a separate portion of the pigment as follows: To 1 g. of the sample in a covered beaker, add 40 cc. of HCl (1:1) and boil gently for 5 to 10 minutes. Wash off cover and evaporate to dryness. Moisten the residue with a few drops of HCl, add about 50 cc. of hot water, boil a few minutes, filter hot through paper, and wash with hot water until washings give no test for lead. (If the sample contains no insoluble matter, the filtration is omitted.) To the filtrate add 20 cc. of H_2SO_4 (sp. gr. 1.84) and evaporate until dense white fumes of H_2SO_4 are copiously evolved. Allow to cool, but not below 60° C., and then add slowly 50 cc. of water while the solution is agitated. Heat to boiling for several minutes in

[1] *U. S. Geological Survey Bulletin 700*, p. 33 (1919).

order to insure complete solution of antimony sulfate. Allow the $PbSO_4$ to settle out until the supernatant liquid is clear, not letting the temperature fall below 60° C. If the liquid does not clear quickly it must be heated longer. When clear, pour the solution through a weighed porcelain Gooch crucible with asbestos mat, decanting the solution as completely as possible without allowing more than a very small amount of $PbSO_4$ to go over into the crucible. Now add 10 cc. more of H_2SO_4 (sp. gr. 1.84) to the $PbSO_4$ in the original beaker, and boil for several minutes. Cool, add slowly 30 cc. of water, and again heat to boiling for a few minutes; allow the solution to cool to about 60° C. and completely transfer the $PbSO_4$ to the Gooch crucible. Wash with "lead acid" (see "Reagents") to remove soluble sulfates and finally wash free of acid with dilute alcohol (equal parts of ethyl alcohol or denatured alcohol and water). Dry in an oven at 105 to 110° C., or, ignite gently in a radiator or muffle. Calculate to PbO. Or, determine as chromate as described below.

If soluble compounds of barium or calcium are present, $BaSO_4$ and $CaSO_4$ will be included with the $PbSO_4$. If soluble SiO_2 is present, it will also be included with the $PbSO_4$. In such cases, the $PbSO_4$ precipitate after washing with dilute alcohol may be dissolved in acid ammonium acetate (see "Reagents") and the lead determined as $PbCrO_4$, as described below. For ordinary work, the amount of $BaSO_4$ dissolved by the acetate treatment may be disregarded.

If the pigment contains no soluble antimony, barium, or calcium compounds, the lead may be determined directly on the original pigment, as follows: To 1 g. of the sample in a covered beaker, add 25 cc. of HNO_3 (1:1), and boil gently a few minutes. Wash off cover, evaporate to dryness on a steam bath, moisten with HNO_3, add hot water, and heat a few minutes. Filter and wash with hot water until washings are lead-free. Add 10 to 20 cc. of H_2SO_4 (1:1) to the clear solution, evaporate and determine lead as $PbSO_4$, as above described.

In the absence of soluble compounds of antimony, iron, aluminum, and barium, the following procedure may be used: Treat 1 g. of the original pigment with 25 cc. of HNO_3 (1:1) and proceed as above. To the clear solution, diluted to 200 cc. add NH_4OH in slight excess, acidify with acetic acid, and add 4 to 6 cc. more of this acid; heat to boiling and add 10 to 15 cc. of a 10-per-cent solution of $K_2Cr_2O_7$. Heat until the yellow precipitate assumes an orange color, let settle and filter on a weighed Gooch crucible, wash by decantation until the washings are colorless, finally transferring all of the precipitate. Then wash with 95-per-cent ethyl alcohol and then with ether; dry to constant weight at 110° C., cool, and weigh $PbCrO_4$. Calculate to PbO.

Antimony Oxide.—Transfer 0.3 g. of a straight antimony oxide pigment, or 0.5 g. of a mixed pigment, to a 500-cc. Pyrex Erlenmeyer flask, add 15 cc. of water and 25 cc. of concentrated HCl (sp. gr. 1.19). Cover with a watch glass, warm on the steam bath 10 to 15 minutes to dissolve the antimony oxide, wash off cover, add 250 cc. of water, and 15 cc. of concentrated H_2SO_4 (sp. gr. 1.84). Boil 2 minutes, cool to 10 to 15° C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution (see "Reagents"). Calculate to Sb_2O_3.

The above procedure gives only the antimony in the *ous* condition. The following method gives the total antimony (*ous* and *ic* forms): Transfer 0.3 g. of a straight antimony oxide pigment, or 0.5 g. of a mixed pigment, to a 500-cc. Pyrex Erlenmeyer flask, add 15 cc. of H_2SO_4 (sp. gr. 1.84), 10 g. of K_2SO_4, and a 9-cm. filter paper (to furnish carbon to act as a reducing agent). Place a funnel in the neck of the flask, and heat until the solution becomes colorless. Cool, wash off the funnel, dilute to 250 cc. with water, add 20 cc. of concentrated HCl, and boil 2 minutes; cool to 10 to 15° C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution. For special work, after digesting, dilute to 100 cc. with water, add 1 to 2 g. of Na_2SO_3, and boil until all the SO_2 is expelled. This is shown when no blue color is obtained with starch-iodate paper (see "Reagents"); the volume will be reduced about one-half. Dilute to 250 cc. with water, add 20 cc. of HCl (sp. gr. 1.19), and complete the determination as described. Calculate total Sb to Sb_2O_3.

Antimony Oxide (in the presence of appreciable amounts of iron).—Treat 1 g. of the mixed pigment, or 0.3 g. of a straight antimony oxide pigment, in a covered 250-cc. beaker with 5 cc. of water and 20 cc. of HCl (sp. gr. 1.19); heat on the steam bath for 15 minutes, cool, wash off cover, add 3 g. of tartaric acid and 100 cc. of hot water, and digest a few minutes. Filter, catching the filtrate in a 500-cc. Pyrex Erlenmeyer flask; wash thoroughly with hot water, dilute to 300 cc. with hot water, and pass in H_2S until the precipitation is complete. (If the sample contains no insoluble matter, disolve directly in a 500-cc. Pyrex Erlenmeyer flask, add tartaric acid, dilute, and pass in H_2S.) Filter, wash with water containing H_2S until free from HCl, return paper and precipitate to the Erlenmeyer flask, add 15 cc. of H_2SO_4 (sp. gr. 1.84) and 10 g. of K_2SO_4, place a funnel in the neck of the flask, and heat until the solution is colorless. Cool, wash off the funnel, carefully dilute the solution to 100 cc. with water, add about 1 g. of Na_2SO_3, and boil until all of the SO_2 is expelled. (Solution will be reduced to less than one-half its former volume.) This is shown when no blue color is obtained with starch-iodate paper (see "Rea-

gents"). Dilute to about 250 cc. with water, add 20 cc. of HCl (sp. gr. 1.19), boil for 2 or 3 minutes, cool to about 10° C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution (see "Reagents"). Calculate the total antimony to Sb_2O_3.

Soluble Barium.—Boil the combined filtrate and washings, reduced in volume by evaporation if need be, from the PbS precipitate (Total Lead) to expel H_2S. Add a slight excess of H_2SO_4 (1:4) over the amount required to precipitate the barium, heat to boiling, let stand on a steam bath about one hour, filter on a weighed Gooch crucible, wash with hot water, dry, ignite, cool, and weigh $BaSO_4$ (Notes 5 and 8). Calculate to BaO.

Alumina (Fe_2O_3, TiO_2, P_2O_5).—Boil the filtrate from the PbS to expel H_2S, add a few drops of HNO_3, and continue the boiling a few minutes to oxidize any iron that may be present. In case soluble barium was present, use the filtrate from that determination. To the solution containing at least 5 g. of NH_4Cl per 200 cc. of solution, or an equivalent amount of HCl, add a few drops of methyl red (0.2-per-cent alcoholic solution) and heat just to boiling. Carefully add dilute NH_4OH drop by drop until the color of the solution changes to a distinct yellow. Boil the solution for one to two minutes and filter at once. Wash the precipitate thoroughly with hot 2-per-cent NH_4Cl solution (Note 9). Ignite the precipitate, cool, and weigh as Al_2O_3 (Note 10).

Total Zinc.—(*a*) To the combined filtrate and washings from the alumina precipitate, add sufficient NH_4Cl to give 5 g. per 100 cc. of solution, and then add 1 g. of ammonium acetate.[1]

Render slightly acid with acetic acid and pass in a current of H_2S to saturation. Allow the precipitate to settle completely, filter on paper, and wash with a 2-per-cent solution of acetic acid saturated with H_2S. Transfer the precipitate and filter to the vessel in which the precipitation was effected, add 30 cc. of water and 10 cc. of concentrated HCl, heat until all zinc is in solution, add 200 cc. of water and a small piece of litmus paper; add strong NH_4OH until slightly alkaline, render just acid with HCl, then add 3 cc. of concentrated HCl, heat nearly to boiling, and titrate with standard potassium ferrocyanide solution as in standardizing that solution (see "Reagents").

(*b*) Zinc may be determined directly on the original sample as follows (Note 11): Weigh accurately about 1 g. (or an amount that will give a burette reading approximately equal to that obtained in the standardization) of the pigment, transfer to a 400-cc. beaker, add 30 cc. of HCl (1:2), boil a few minutes, add 200 cc. of water and

[1] F. A. Gooch, "Representative Procedures in Quantitative Chemical Analysis," 1st Ed., p. 107.

a small piece of litmus paper; add strong NH_4OH until slightly alkaline, render just acid with HCl, then add 3 cc. of concentrated HCl, heat nearly to boiling, and titrate with standard $K_4Fe(CN)_6$ solution as in standardizing that solution (see "Reagents").

(*c*) When iron is present, total zinc may be determined directly on the original sample as follows (Note 11): Weigh accurately about 1 g. (or an amount that will give a burette reading approximately equal to that obtained in the standardization) of the pigment, transfer to a 250-cc. beaker, moisten with alcohol, add 30 cc. of HCl (1:2), boil for 2 or 3 minutes, and add about 100 cc. of water. Add about 2 g. of NH_4Cl, make slightly alkaline with NH_4OH, heat to boiling, let settle on steam bath, filter into a 400-cc. beaker and wash the residue once with hot water. Remove the 400-cc. beaker and pour dilute HCl on the residue, catching the filtrate therefrom in the 250-cc. beaker, wash a few times with hot water. Add to this filtrate 1 g. of NH_4Cl and make slightly alkaline with NH_4OH, boil, let settle, filter on paper used for first filtration, and wash thoroughly with hot water, catching the filtrate and washings in the 400-cc. beaker containing the first filtrate. Add a small piece of litmus paper, acidify with HCl, add 3 cc. of concentrated HCl, heat nearly to boiling, and titrate with standard $K_4Fe(CN)_6$ as above.

(*d*) With pigments containing ZnO and ZnS the ZnO may be determined as follows: Weigh accurately 2.5 g. of the pigment, transfer to a 250-cc. graduated flask, moisten with a few drops of alcohol, add about 200 cc. of 2 to 3-per-cent acetic acid, shake vigorously and let stand for 30 minutes at room temperature, shaking once every 5 minutes. Then let stand at room temperature at least 5 hours, preferably overnight. Fill to the mark with 2 to 3-per-cent acetic acid, mix, filter through a dry paper, discard the first 25 cc. and transfer 100 cc. of the filtrate (corresponding to 1 g.) to a 400-cc. beaker. To the clear solution add 30 cc. of HCl (1:2), 100 cc. of H_2O, and a small piece of litmus paper; add strong NH_4OH until slightly alkaline, render just acid with HCl, then add 3 cc. of concentrated HCl, heat nearly to boiling, and titrate with $K_4Fe(CN)_6$ as above. Calculate the percentage of ZnO (any $ZnCO_3$ or $ZnSO_4$ is included in the ZnO). Subtract this result from the percentage of total Zn as ZnO, and calculate the difference to ZnS.

Soluble Calcium.—Heat the united filtrate and washings, reduced in volume if need be, from the ZnS precipitate, to boiling, add 1 cc. of NH_4OH and an excess of a hot saturated ammonium oxalate solution. Continue the boiling until the precipitate becomes granular; let stand about one hour, filter, and wash with hot water. Ignite, cool,

and weigh as CaO (Notes 5, 12, 13); or, place the beaker in which the precipitation was made under the funnel, pierce the apex of the filter with a stirring rod and wash the precipitate into the beaker with hot water, pour warm dilute H_2SO_4 (1:4) through the paper and wash a few times. Add about 30 cc. of dilute H_2SO_4 (1:4), dilute to about 250 cc., heat to 90° C. and titrate at once with standard (0.1 N) $KMnO_4$ solution (the temperature of the solution should not be below 60° C. when the end point is reached. See "Reagents"). Calculate to CaO (Notes 5, 12, 13). (The Fe value of $KMnO_4 \times 0.502$ = CaO value.)

Soluble Magnesium.—Acidify the filtrate from the calcium precipitate with HCl, add 10 cc. of a saturated solution of $Na(NH_4)HPO_4$ and NH_4OH drop by drop, with constant stirring. When the crystallin $(NH_4)MgPO_4$ has formed, add 5 cc. excess of NH_4OH. Allow the solution to stand in a cool place for not less than 4 hours, preferably overnight (Note 14); filter and wash with water containing 2.5 per cent of NH_3. Dissolve the precipitate in a small quantity of hot dilute HCl, dilute the solution to about 100 cc. with water, add 1 cc. of a saturated solution of $Na(NH_4)HPO_4$ and NH_4OH drop by drop, with constant stirring, until the precipitate is again formed as described, and then add 5 cc. excess of NH_4OH. Let the precipitate stand in a cool place for not less than 2 hours, filter on a Gooch crucible, wash with water containing 2.5 per cent of NH_3, ignite, cool, and weigh as $Mg_2P_2O_7$ (Note 15). Calculate to MgO.

Carbon Dioxide.—Determine by evolution with dilute acid and absorption in soda lime or KOH solution. The method given in U. S. Geological Survey Bulletin 700, p. 218, shows a convenient apparatus for carrying out this determination. Use from 1 to 2 g. of the pigment, depending upon the probable CO_2 content, following the method for the determination of carbon dioxide in lime described in the Tentative Methods of Chemical Analysis of Limestone, Quicklime and Lime Products (Serial Designation: C 25 – 25 T) of the American Society for Testing Materials[1] (Note 16).

Total Soluble Sulfur Compounds (Note 5).—Treat 1 g. of the pigment in a 400-cc. beaker with 10 cc. of H_2O, 10 cc. of strong HCl saturated with bromine, and 5 g. of NH_4Cl, digest (covered) on a steam bath for 5 minutes, dilute with hot water to about 200 cc., boil for 5 minutes, filter to separate any insoluble matter, and wash thoroughly with hot water. Nearly neutralize the clear solution in a covered beaker with NaOH solution, complete the neutralization with dry Na_2CO_3 and add about 2 g. more of this reagent. Boil 10 to 15

[1] See p. 233.

minutes, wash off cover, let settle, filter, and wash with hot water. Re-dissolve the precipitate in HCl (1:1), reprecipitate with Na_2CO_3 as above, filter, and wash thoroughly with hot water. Acidify the united filtrates with HCl, adding about 1 cc. in excess. Boil to expel bromine, and to the clear boiling solution add slowly with stirring an excess of a 10-per-cent $BaCl_2$ solution. Let stand on a steam bath for at least 1 hour, filter on a weighed Gooch crucible, wash thoroughly with boiling water, dry, ignite at a dull red heat, cool, and weigh as $BaSO_4$. This will include soluble sulfates, SO_3 formed from SO_2 and the SO_3 that is formed from sulfide sulfur (Note 8).

Soluble Sulfate (Note 5).—Treat 1 g. of the pigment with 10 cc. of H_2O and 10 cc. of concentrated HCl and 5 g. of NH_4Cl. Boil until H_2S is expelled, adding more HCl (1:1) if necessary; dilute with hot water to about 200 cc., boil for 5 minutes, filter to separate any insoluble matter, and wash thoroughly with hot water. Nearly neutralize the clear solution with NaOH solution and make a double precipitation with Na_2CO_3, as in preceding method, finally weighing as $BaSO_4$, as described above (Note 8).

Sulfide Sulfur[1] (Note 17).—Place 0.5 to 1 g. of the pigment in a flask with about 10 g. of "feathered" or mossy zinc, add 50 cc. of water; insert a stopper carrying a separatory funnel and an exit tube. Run in 50 cc. of concentrated HCl from the funnel, having previously connected the exit tube to two absorption flasks in series; the first flask contains 100 cc. of alkaline lead-nitrate solution (see "Reagents"), the second flask, 50 cc. of the same solution as a safety device. After all of the acid has run into the evolution flask, heat slowly, finally boiling until the first appearance of steam in the first absorption flask. Disconnect, let the lead sulfide settle, filter, wash with cold water, then with hot water till neutral to litmus paper and washings give no test for lead. Dissolve the PbS precipitate in hot, dilute HNO_3, and determine the lead as $PbSO_4$. Calculate to S. For very rapid work, the evolved H_2S may be absorbed in an ammoniacal $CdCl_2$ or $ZnSO_4$ solution (see "Reagents") contained in 2 flasks connected in series, the contents of the absorption flasks washed into a vessel with cold water and diluted to about one liter, acidified with concentrated HCl and titrated with standard potassium iodate solution (see "Reagents"), using starch indicator (see "Reagents").

Sulfur Dioxide (Note 18).—Transfer 10 g. of the pigment to a suitable flask, insert a stopper fitted with a separatory funnel and a spray trap delivery tube (Note 19), and attach the latter to a con-

[1] Evolution Method of W. G. Scott, "White Paints and Painting Materials," p. 257; see also Blair, "The Chemical Analysis of Iron."

denser. Place about 150 cc. of HCl (1:3) in the funnel, the stopcock being closed (Note 20), connect the other end of the condenser with a delivery tube which passes through a 2-hole stopper and extends nearly to the bottom of an absorption flask; through the other hole of the stopper connect a tube or flask to serve as a safety device. Place 25 cc. of 0.05 *N* iodine solution (see "Reagents") in the absorption flask (dilute with water if need be) and 20 cc. of 10-per-cent KI solution in the safety tube; fit stopper in the absorption flask. Open the stopcock and allow the acid to slowly enter the flask. Before all of the acid is admitted, air (washed with NaOH solution) is forced through the top of the separatory funnel (about 2 bubbles per second in the KI solution). Boil the solution 3 minutes with the air passing through, then remove the source of heat and pass air through for 30 minutes. Disconnect the absorption vessels, wash the KI solution into the iodine solution, and titrate at once with 0.05 *N* $Na_2S_2O_3$ solution, using starch indicator. Run a blank determination in exactly the same manner except for the omission of the pigment. Subtract this figure from the previous one and calculate the final result to SO_2 (1 cc. 0.05 *N* I = 0.0016 g. SO_2).

Matter Soluble in Water.—Transfer 2.5 g. of the pigment to a graduated 250-cc. flask, add 100 cc. of water, boil for 5 minutes, cool, fill to mark with water, mix, and allow to settle. Pour the supernatant liquid through a dry filter paper and discard the first 20 cc. Then evaporate 100 cc. of the clear filtrate to dryness in a weighed dish, heat for one hour at 105 to 110° C., cool, and weigh. Calculate the percentage. The nature of this may be determined by further examination, as the percentages of SO_3 and CaO may be indicative.

NOTES

1. A convenient apparatus for this determination is shown in Fig. 1 (*b*) of the Standard Method of Test for Water in Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[1]

2. In the absence of much gum or resin the iodine number gives the best, though doubtful, indication of linseed oil. The fatty acids may be tested for rosin and examined for unsaponifiable matter, following the methods given in the books on paints analysis. In some cases it may be desirable to let a portion of the well-mixed paint stand until the pigment settles out (or settle by means of a centrifuge), decant the vehicle, distil off the thinner, and examine the nonvolatile vehicle.

3. On an extracted and dried pigment, this determination is of little value. If the original paint contained gypsum, a part of the combined H_2O of the latter will be driven off in the drying of the extracted pigment and in the "moisture" determination.

4. This determination may serve as a rough or approximate check in many cases on the CO_2, H_2O, etc.

[1] 1924 Book of A.S.T.M. Standards.

5. If the original sample contained $BaCO_3$ and $PbSO_4$, $CaSO_4$ or other soluble sulfate, the soluble Ba will form with the soluble sulfate a precipitate of $BaSO_4$ which will be determined as "insoluble matter." If the sample contained $SrSO_4$ or $SrCO_3$, some $SrSO_4$ may be counted as $BaSO_4$, some Sr will count as soluble barium, and some may be counted as CaO. This element is not separated, as it probably will not be encountered, or will be present as an impurity in the Ba and Ca compounds.

6. Any soluble Al_2O_3 (Fe_2O_3) and in most cases MgO, and sometimes some CaO, come from the siliceous pigment used. MgO generally denoted the presence of asbestine.

7. It is not possible to determine the amount of basic lead carbonate and lead sulfate when carbonates or soluble sulfates of other metals, such as calcium, are present. Also neither basic lead carbonate nor basic lead sulfate are definite compounds.

8. This will include any $BaSO_4$ that may have been dissolved as such. The weighed precipitate should be tested for $CaSO_4$, and if present, it should be removed by treating with hot dilute HCl, filtering, washing, igniting, and again weighing.

9. For very accurate work, or when the precipitate is large, the precipitate should be dissolved in HCl (1:1) and the precipitation repeated.

10. This precipitate may also contain Fe_2O_3, TiO_2, and P_2O_5.

11. If the sample contains antimony, it should be precipitated by H_2S in the hot acid solution, filtered off, washed, and the filtrate neutralized, etc., for zinc. The H_2S precipitate may also contain PbS. If no sulfide separation is made, any cadmium present will be counted as zinc.

12. Care must be exercised in this washing, as 1000 cc. of boiling water will dissolve over 0.01 g. of CaC_2O_4.

13. For more accurate work, the CaC_2O_4 precipitate should be ignited, cooled, cautiously moistened with water, redissolved in HCl and the solution diluted to 100 cc. Then NH_4OH should be added in slight excess, the liquid boiled, and filtered and washed if a precipitate appears. Then reprecipitate the Ca with NH_4OH and $(NH_4)_2C_2O_4$ as above, filter, wash, ignite, cool, and weigh; or, titrate as described.

14. The less the amount of magnesium present, the longer the precipitate must be allowed to settle.

15. If the sample contained manganese, it will be caught in large part with the $Mg_2P_2O_7$. If desired, Mn may be determined by dissolving the $Mg_2P_2O_7$ in HNO_3 and applying the bismuthate method.

16. If the sample is high in sulfide, *e. g.*, contains a high percentage of lithopone, grind 1 to 2 g. of the pigment with dry $K_2Cr_2O_7$, transfer to the evolution flask, add 50 cc. of water, and run in H_2SO_4 (1:1) from the separatory funnel. Or, place at the front of the purifying and drying train a tube containing acidified $CuSO_4$ solution, $KMnO_4$ solution, or CrO_3 solution.

17. The percentage of sulfide sulfur can be calculated from the percentages of total zinc and zinc soluble in 2 to 3 per cent acetic acid, assuming the sulfide to be ZnS. See Method (*d*) under determination of zinc.

18. This method is not applicable in the presence of sulfides decomposable under the conditions given.

19. A Knorr CO_2 apparatus is very convenient. In this case, the vertical condenser may be connected with an absorption tower containing the iodine solution, followed by the KI solution in a suitable tube.

20. To minimize, if not eliminate, any possible oxidation by the air, add about

1 g. (in one piece) of $NaHCO_3$ to the evolution flask, then add the acid directly to the flask, omitting the separatory funnel and the current of air. Boil the solution until about 50 cc. of distillate has passed over.

CALCULATIONS

The calculation of the component pigments of a mixed or combination pigment may be a somewhat difficult matter. Certain assumptions must be made, depending upon the complexity of the mixed pigment, as to the composition or formulas of component pigments and as to the manner in which the acidic and basic radicles are combined. Add any Al_2O_3 (Fe_2O_3) found in the soluble portion to the siliceous matter and report the sum as "Insoluble siliceous matter," unless the soluble Al is high; in this case, an aluminate is probably present, and the Al_2O_3 should be reported as Al_2O_3. If a small amount of soluble Mg is found, it should also be added to the siliceous matter. If the soluble Mg is high, the presence of $MgCO_3$ is indicated, and the MgO is calculated to $MgCO_3$ as pointed out below. The insoluble siliceous matter reported should be based on the weight obtained on drying the total insoluble matter at 105° C. if the combined H_2O contained therein is to be considered.

In the absence of ZnS or TiO_2, report $BaSO_4$ as $BaSO_4$. If ZnS is present, calculate the $BaSO_4$ equivalent by multiplying by 2.85; report sum of $ZnS+BaSO_4$ as "lithopone." If TiO_2 is present, calculate the $BaSO_4$ equivalent by multiplying by 3.17; report sum of TiO_2+BaSO_4 as "titanium pigment." Report residual $BaSO_4$ as $BaSO_4$. If TiO_2 is present and $BaSO_4$ is absent or is present in a smaller amount than would be indicated by the above factor, then report TiO_2 as TiO_2, and $BaSO_4$ as $BaSO_4$. If $CaCO_3$, $CaSO_4$, $BaCO_3$, and $MgCO_3$ are absent calculate CO_2 to basic carbonate white lead, $(PbCO_3)_2 \cdot Pb(OH)_2$, and soluble SO_3 to $PbSO_4$. Any excess of Pb is calculated to PbO, added to the $PbSO_4$, and the sum reported as basic lead sulfate; or, multiply the sum of $PbSO_4+PbO$ by 0.058 to obtain the ZnO; add this result to the $PbSO_4+PbO$ and report as basic sulfate white lead. (The ZnO factor is based on the assumption that the average composition of commercial basic sulfate white lead is: 78.5 per cent $PbSO_4$, 16.0 per cent PbO, and 5.5 per cent ZnO.) Lead oxide (PbO) should not be reported except in the presence of $PbSO_4$; unless the entire analysis is reported in the elementary or oxide form.

If the sample contains CO_2 but no soluble SO_3, calculate total Pb to basic carbonate white lead, $(PbCO_3)_2 \cdot Pb(OH)_2$; calculate residual CO_2 to $CaCO_3$, then to $BaCO_3$ and $MgCO_3$ if soluble Ba and Mg should be present in sufficient amounts to indicate the presence of

these carbonates. The CO_2 result will be an index of this. A small amount of residual CaO is probably from the sliceous matter and should be added to the insoluble siliceous matter.

A small amount of soluble Ba may be from the $CaCO_3$ used or may be due to the solubility of $BaSO_4$ if this compound is present in the original pigment. This Ba may be calculated to $BaSO_4$ and added to the $BaSO_4$ found in the insoluble matter.

If the sample contains soluble SO_3 but no CO_2, calculate CaO to $CaSO_4$ or $CaSO_4 \cdot 2H_2O$; residual SO_3 to $PbSO_4$; add residual PbO to. $PbSO_4$ and report sum as basic lead sulfate; or, multiply $PbSO_4$+PbO by 0.058 and add the result to the $PbSO_4$+PbO, and report the total as basic sulfate white lead.

If the sample contains $CaCO_3$ ($MgCO_3$, $BaCO_3$) and also basic sulfate white lead, or $CaSO_4$ and basic carbonate white lead; or a mixture of these; it is not possible to determine or calculate the amount of $PbCO_3$ or $PbSO_4$ with any degree of certainty (Notes 3 and 5). The presence of appreciable amounts of CaO and SO_3 in the water-soluble matter indicates the probable presence of $CaSO_4$ in the original pigment. The following arbitrary calculations may be made; calculate water-soluble SO_3 to $CaSO_4$ or $CaSO_4 \cdot 2H_2O$, subtract this SO_3 from total soluble SO_3 and calculate the remainder to $PbSO_4$; calculate residual CaO to $CaCO_3$, and then residual CO_2 to $(PbCO_3)_2 \cdot Pb(OH)_2$. If there is an excess of CO_2, calculate to $MgCO_3$ or $BaCO_3$, if the amounts of soluble Mg and Ba indicate the probable presence of these carbonates. Add residual PbO to $PbSO_4$ and calculate, as above, to basic sulfate white lead. The procedure followed by the Federal Specifications Board should be noted.[1]

Report total antimony as Sb_2O_3.

Calculate sulfide sulfur to ZnS, subtract the Zn equivalent to the S from the total Zn, then subtract the Zn required for the basic sulfate white lead, and report the remainder as ZnO.

Report moisture, loss on ignition, So_2, and matter soluble in water directly.

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[1] Federal Specifications Board Specification No. 10 for "White Paint and Tinted Paints Made on a White Base, Semipaste and Ready Mixed"; Bureau of Standards *Circular No. 89*, 2d Ed., p. 2: "The total lead dissolved by dilute acetic acid and hot acid, ammonium acetate, weighed as lead sulfate, and this weight multiplied by the factor 0.883 shall be considered white lead. (It is not possible to determine the amount of lead carbonate and lead sulfate when carbonates or sulfates of other metals, such as calcium, are present. Also neither basic lead carbonate nor basic lead sulfate are definite compounds. The factor to convert $PbSO_4$ to $(PbCO_3)_2Pb(OH)_2$ is 0.854, to convert $PbSO_4$ to $PbSO_4 \cdot PbO$ is 0.868, and to convert $PbSO_4$ to $(PbSO_4)_2 \cdot PbO$ is 0.913. The arbitrary factor used under this specification is the mean of the largest and smallest of these three factors.)"

TENTATIVE METHOD OF TEST

FOR

DISTILLATION OF GASOLINE, NAPHTHA, KEROSINE, AND SIMILAR PETROLEUM PRODUCTS[1]

Serial Designation: D 86 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1923, 1924, 1925.

APPARATUS

1. *Flask.*—The Standard 100-cc. Engler flask is shown in Fig. 1, the dimensions and allowable tolerance being as follows: **Flask.**

	Centimeters	Inches	Tolerances, cm
Diameter of bulb, outside	6.5	2.56	0.2
" " neck, inside	1.6	0.63	0.1
Length of neck	15.0	5.91	0.4
" " vapor tube	10.0	3.94	0.3
Diameter of vapor tube, outside	0.6	0.24	0.05
" " " " inside	0.4	0.16	0.05
Thickness of vapor tube wall	0.1	0.04	0.05

The position of the vapor tube shall be 9 cm. (3.55 in.) ±3mm. above the surface of the liquid when the flask contains its charge of 100 cc. The tube is approximately in the middle of the neck and set at an angle of 75 deg. (tolerance ±3 deg.) with the vertical.

2. *Condenser.*—The condenser (Fig. 2) consists of a $\frac{9}{16}$-in. (14.29 mm.) OD No. 20 Stubbs Gage seamless brass tube, 22 in. (55.88 cm.) long. It is set at an angle of 75 deg. from the perpendicular and is surrounded with a cooling bath 15 in. long (38.1 cm.), approximately 4 in. (10.16 cm.) wide by 6 in. (15.24 cm.) high. The lower end of the condenser tube is cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to $1\frac{1}{4}$ in. (2.54 to 3.8 cm.) below the top of the graduate when it is in position to receive the distillate. **Condenser.**

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

Shield. 3. *Shield.*—The shield (Fig. 2) is made of approximately 22 gage sheet metal and is 19 in. (48.26 cm.) high, 11 in. (27.94 cm.) long and 8 in. (20.32 cm.) wide, with a door on one narrow side, with two openings, 1 in. (2.54 cm.) in diameter, equally spaced, in each of

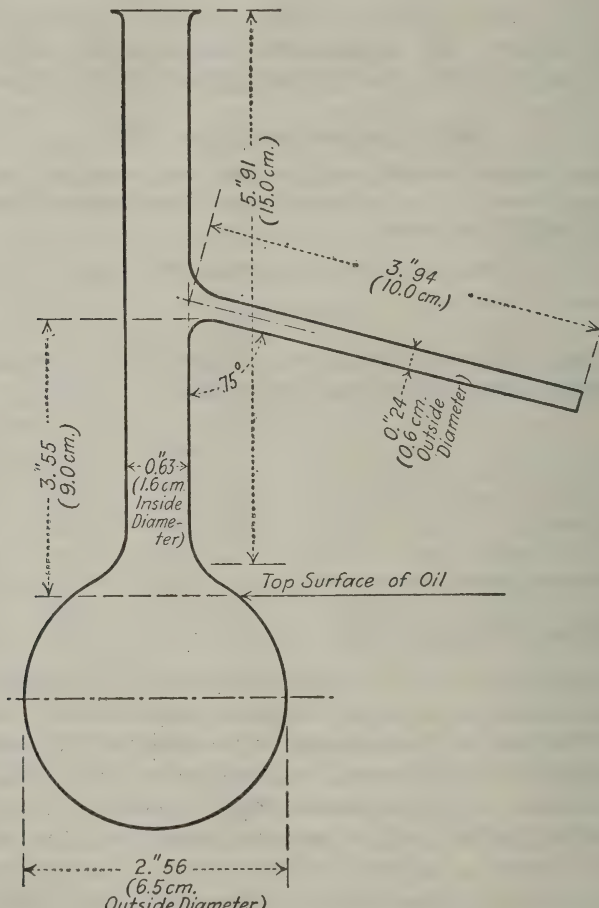

FIG. 1.

the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings are $8\frac{1}{2}$ in. (21.59 cm.) below the top of the shield. There are also three $\frac{1}{2}$-in. (1.27 cm.) holes in each of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield.

4. *Ring Support and Hard Asbestos Boards.*—The ring support is of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and is supported on a stand inside the shield. There are two hard asbestos boards: One 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.) with a hole $1\frac{1}{4}$ in. (3.175 cm.)[1] in diameter in its center, the sides of which shall be perpendicular to the surface; the other, an asbestos board to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These are arranged as **Support.**

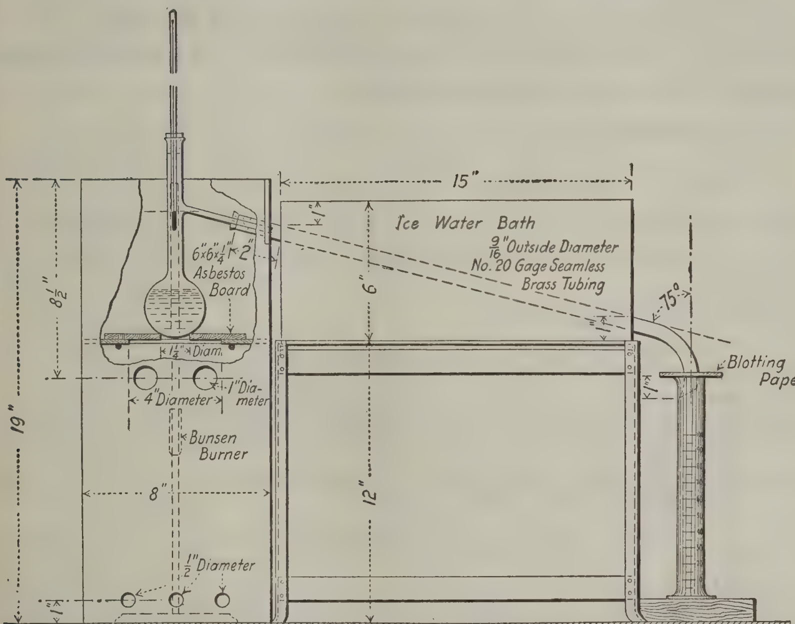

FIG. 2.

follows: The second asbestos board is placed on the ring and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat is applied to the flask only through the $1\frac{1}{4}$-in. (3.175 cm.) opening in the first asbestos board.

5. *Gas Burner or Electric Heater.*— **Heater.**

(*a*) *Gas Burner.*—The burner is so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified below. The flame should never be so large that it spreads over a

[1] When distilling petroleum products having an end point above 470° F. (243.34° C.), the hole in the asbestos board shall be $1\frac{1}{2}$ in. (3.81 cm.) in diameter.

circle of diameter greater than $3\frac{1}{2}$ in. (8.89 cm.) on the under surface of the asbestos board. A sensitive regulating valve is a necessary adjunct, as it gives complete control of heating.

(*b*) *Electric Heater*.—The electric heater, which may be used in place of the gas flame, shall be capable of bringing over the first drop within the time specified below when started cold, and of continuing the distillation at the uniform rate. The electric heater shall be fitted with an asbestos board top $\frac{1}{8}$ to $\frac{1}{4}$ in. (3.175 to 6.35 mm.) thick, having a hole $1\frac{1}{4}$ in. (3.175 cm.) in diameter in the center. When an electric heater is employed, the portion of the shield above the asbestos board shall be the same as with the gas burner but the part below the board may be omitted.

Thermometers.

6. *Thermometers*.—

(*a*) The A.S.T.M. Low-Distillation Thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated in either Centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 300° C. or 30 to 580° F., respectively.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: 0 to 300° C. in 1° C. or 30 to 580° F. in 2° F.

TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).

STEM: Plain front, enamel back, suitable thermometer tubing.
Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).

DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 100 to 110 mm. (3.94 to 4.33 in.).

DISTANCE TO 300° C. OR 572° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. Low Distillation," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.5° C. or 1° F.

STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 300° C. or 572° F.

TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature of 280 to 290° C. or 540 to 560° F. for 24 hours, the accuracy shall be within the limit specified.

CASE: The thermometer shall be supplied in a suitable case on which shall appear The marking: "A.S.T.M. Low Distillation, 0 to 300° C." or "A.S.T.M. Low Distillation, 30 to 580° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

(*b*) The A.S.T.M. High-Distillation Thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated either in Centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 400° C. or 30 to 760° F., respectively.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: 0 to 400° C. in 1° C. or 30 to 760° F. in 2° F.

TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).

DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 25 to 35 mm. (0.98 to 1.38 in.).

DISTANCE TO 400° C. OR 752° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. High Distillation" a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale up to 370° C. or 700° F. when the thermometer is standardized as provided below shall not exceed 1° C. or 2° F.

STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 370° C. or 700° F.

TEST FOR PERMANENCY OF RANGE: After being subjected to a temperature between 360 and 370° C. or 680 and 700° F. for 24 hours, the accuracy shall be within the limit specified.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. High Distillation, 0 to 400° C." or "A.S.T.M. High Distillation, 30 to 760° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

Graduate. 7. *Graduate.*—The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 cc., and the graduated portion shall be not less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) long. It shall be graduated in single cubic centimeters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 cc. The overall height of the graduate shall be not less than $9\frac{3}{4}$ in. (24.8 cm.), nor more than $10\frac{1}{4}$ in. (26.0 cm.). The graduations shall not be in error by more than 1 cc. at any point on the scale.

PROCEDURE

Procedure. 8. (*a*) The condenser bath shall be filled with cracked ice,[1] and enough water added to cover the condenser tube. The temperature shall be maintained between 32 and 40° F. (0 and 4.45° C.).

(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft cloth attached to a cord or copper wire may be used for this purpose.

(*c*) One hundred cubic centimeters of the product shall be measured in the 100-cc. graduated cylinder at 55 to 65° F. (12.78 to 18.33° C.) and transferred directly to the Engler flask. None of the liquid shall be permitted to flow into the vapor tube.

(*d*) The thermometer[2] provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(*e*) The charged flask shall be placed in the $1\frac{1}{4}$-in. (3.175-cm.) opening in the 6 by 6-in. (15.24 by 15.24-cm.) asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.).

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[1] Any other convenient cooling medium may be used.

[2] For products having an initial boiling point of 212° F. (100° C.) or higher, the High-Distillation Thermometer shall be used; for all other products, the Low-Distillation Thermometer shall be used.

(*f*) The graduated cylinder used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 in. (2.54 cm.) but not below the 100-cc. mark. Unless the temperature is between 55 and 65° F. (12.78 and 18.33° C.) the receiving graduate shall be immersed up to the 100-cc. mark in a transparent bath maintained between these temperatures. The top of the graduate shall be covered closely during the distillation with a piece of blotting paper or its equivalent, cut so as to fit the condenser tube tightly.

9. When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 minutes. When the first drop falls from the end of the condenser the reading of the distillation thermometer shall be recorded as the *initial boiling point.* The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation will proceed at a uniform rate of not less than 4 nor more than 5 cc. per minute. The volume of distillate collected in the cylinder shall be observed and recorded, to the nearest 0.5 cc., when the mercury of the thermometer reaches each point that is an even multiple of 25° C. or the Fahrenheit equivalent of this point (50° C., 75° C., 100° C., 125° C., etc., or 122° F., 167° F., 212° F., 257° F., etc.). **Distillation.**

When the liquid residue in the distillation flask is approximately 5 cc. the heat may be increased because of the presence of heavy ends which have relatively high boiling points. However, no further increase of heat shall be applied after this adjustment. The 4 to 5-cc. rate can rarely be maintained from this point to the end of the distillation, but in no case shall the period between the point when approximately 5 cc. of liquid remains in the flask and the end point be more than 5 minutes.

The heating shall be continued until the mercury reaches a maximum and starts to fall consistently. The highest temperature observed on the distillation thermometer shall be recorded as the *maximum temperature* or end point. Usually this point will be reached after the bottom of the flask has become dry.

The total volume of the distillate collected in the receiving graduate shall be recorded as the *recovery*.

The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 cc., measured when cool and the volume recorded as *residue*.

The difference between 100 cc. and the sum of the recovery and the residue shall be calculated and recorded as *distillation loss.*

ACCURACY

Accuracy. 10. With proper care and attention to detail, duplicate results obtained for initial boiling point and maximum temperature, respectively, should not differ from each other by more than 6° F. (3.33° C.).

CORRECTION FOR BAROMETRIC PRESSURE

11. The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute. In such cases the temperature points shall be corrected to 760 mm. (29.92 in.), by the use of the Sydney Young equation, as follows:

For centigrade readings:

$$C_c = 0.00012\ (760-P)\ (273 + t_c)$$

For Fahrenheit readings:

$$C_f = 0.00012\ (760-P)\ (460 + t_f)$$

in which C_c and C_f are, respectively, corrections to be added to the observed temperature t_c or t_f, and P is the actual barometric pressure in millimeters of mercury.

The following table is a convenient approximation of the corrections as calculated by the above equation.

Temperature Range		Correction[1] per 10-mm. Difference in Pressure,	
deg. Cent.	deg. Fahr.	deg. Cent.	deg. Fahr.
10 – 30	50 – 86	0.35	0.63
30 – 50	86 – 122	0.38	0.68
50 – 70	122 – 158	0.40	0.72
70 – 90	158 – 194	0.42	0.76
90 – 110	194 – 230	0.45	0.81
110 – 130	230 – 266	0.47	0.85
130 – 150	266 – 302	0.50	0.89
150 – 170	302 – 338	0.52	0.94
170 – 190	338 – 374	0.54	0.98
190 – 210	374 – 410	0.57	1.02
210 – 230	410 – 446	0.59	1.06
230 – 250	446 – 482	0.62	1.11
250 – 270	482 – 518	0.64	1.15
270 – 290	518 – 554	0.66	1.19
290 – 310	554 – 590	0.69	1.24
310 – 330	590 – 626	0.71	1.28
330 – 350	626 – 662	0.74	1.32
350 – 370	662 – 698	0.76	1.37
370 – 390	698 – 734	0.78	1.41
390 – 410	734 – 770	0.81	1.45

[1] To be added in case barometric pressure is below 760 mm.; to be subtracted in case barometric pressure is above 760 mm.

TENTATIVE METHOD OF TEST
FOR
DISTILLATION OF NATURAL GAS GASOLINE[1]

Serial Designation: D 216 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

I. APPARATUS

1. *Flask.*—The standard 100-cc. Engler flask is shown in Fig. 1, the dimensions and allowable tolerance being as follows: **Flask.**

	Centimeters	Inches	Tolerances, cm.
Diameter of bulb, outside	6.5	2.56	0.2
Diameter of neck, inside	1.6	0.63	0.1
Length of neck	15.0	5.91	0.4
Length of vapor tube	10.0	3.94	0.3
Diameter of vapor tube, outside	0.6	0.24	0.05
Diameter of vapor tube, inside	0.4	0.16	0.05
Thickness of vapor tube wall	0.1	0.04	0.05

The position of the vapor tube shall be 9 cm. (3.55 in.) ±3 mm. above the surface of the liquid when the flask contains its charge of 100 cc. The tube is approximately in the middle of the neck and set at an angle of 75 deg. (tolerance ±3 deg.) with the vertical.

2. *Condenser.*—The condenser (Fig. 2) consists of a $\frac{9}{16}$-in. (14.29-mm.) OD No. 20 Stubbs Gage seamless brass tube, 22 in. (55.88 cm.) long. It is set at an angle of 75 deg. from the perpendicular and is surrounded with a cooling bath 15 in. long (38.1 cm.) approximately 4 in. (10.16 cm.) wide by 6 in. (15.24 cm.) high. The lower end of the condenser tube is cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to $1\frac{1}{4}$ in. (2.54 to 3.8 cm.) below the top of the graduate when it is in position to receive the distillate. **Condenser.**

3. *Shield.*—The shield (Fig. 2) is made of approximately No. 22 gage sheet metal and is 19 in. (48.26 cm.) high, 11 in. (27.94 cm.) long and 8 in. (20.32 cm.) wide, with a door on one narrow side, with two **Shield.**

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

openings, 1 in. (2.54 cm.) in diameter equally spaced, in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings are 8½ in. (21.59 cm.) below the top of the shield. There are also three ½ in. (1.27 cm.) holes in each

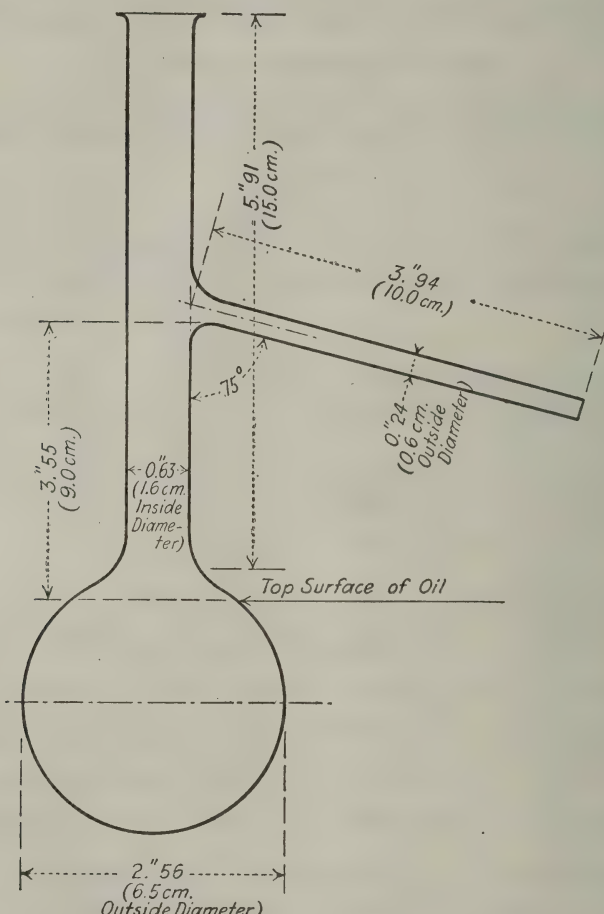

FIG. 1.

of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield.

Support. 4. *Ring Support and Hard Asbestos Boards.*—The ring support is of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and is supported on a stand inside the shield. There are two hard

asbestos boards: one 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.), with a hole $1\frac{1}{4}$ in. (3.175 cm.) in diameter in its center, the sides of which shall be perpendicular to the surface; the other, to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These are arranged as follows: The second asbestos board is placed on the ring, and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat is applied to

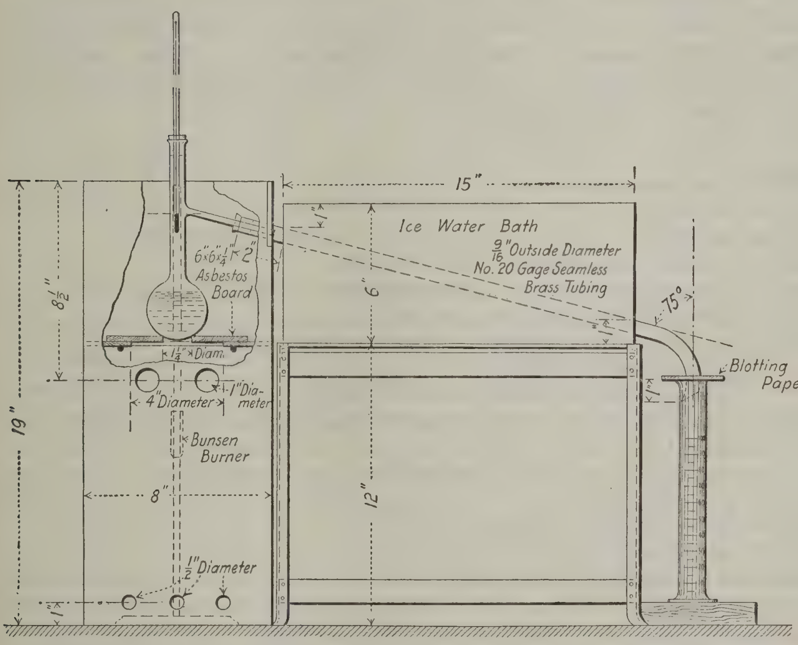

FIG. 2.

the flask only through the $1\frac{1}{4}$ in. (3.175 cm.) opening in the first asbestos board.

5. *Gas Burner or Electric Heater.*

(*a*) *Gas Burner.*—The burner is so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified below. The flame should never be so large that it spreads over a circle of a diameter greater than $3\frac{1}{2}$ in. (8.89 cm.) on the under surface of the asbestos board. A sensitive regulating valve is a necessary adjunct, as it gives complete control of heating. **Heater.**

(*b*) *Electric Heater.*—The electric heater, which may be used in place of the gas flame, shall be capable of bringing over the first drop within the time specified below when started cold, and of continuing the distillation at the uniform rate. The electric heater shall be fitted with an asbestos board top $\frac{1}{8}$ to $\frac{1}{4}$ in. (3.175 to 6.35 mm.) thick, having a hole $1\frac{1}{4}$ in. (3.175 cm.) in diameter in the center. When an electric heater is employed, the portion of the shield above the asbestos board shall be the same as with the gas burner but the part below the board may be omitted.

Thermometer.

6. *Thermometer.*—The A.S.T.M. Low-Distillation Thermometer shall conform to the following requirements. These specifications cover a total-immersion thermometer graduated in either Centigrade or Fahrenheit degrees, as specified, the ranges being 0 to 300° C. or 30 to 580° F., respectively.

Type: Etched stem, glass.

Liquid: Mercury.

Range and Subdivision: 0 to 300° C. in 1° C. or 30 to 580° F. in 2° F.

Total Length: 378 to 384 mm. (14.88 to 15.12 in.).

Stem: Plain front, enamel back, suitable thermometer tubing.
Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

Bulb: Corning normal or equally suitable thermometric glass.
Length, 10 to 15 mm. (0.39 to 0.59 in.).
Diameter, 5.0 to 6.0 mm. (0.20 to 0.24 in.).

Distance to 0° C. or 32° F. Line from Bottom of Bulb: 100 to 110 mm. (3.94 to 4.33 in.).

Distance to 300° C. or 572° F. Line from Top of Thermometer: 30 to 45 mm. (1.18 to 1.77 in.).

Filling above Mercury: Nitrogen gas.

Top Finish: Glass ring.

Graduation: All lines, figures, and letters clear cut and distinct. The first and each succeeding 5° C. or 10° F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

Immersion: Total.

Special Marking: "A.S.T.M. Low Distillation," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

Scale Error: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.5° C. or 1° F.

Standardization: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 50° C. or 100° F. up to 300° C. or 572° F.

Test for Permanency of Range: After being subjected to a temperature of 280 to 290° C. or 540 to 560° F. for 24 hours, the accuracy shall be within the limit specified.

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Low Distillation, 0 to 300° C." or "A.S.T.M. Low Distillation, 30 to 580° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the overall length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

7. *Graduate.*—The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 cc., and the graduated portion shall be not less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) long. It shall be graduated in single cubic centimeters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up, at intervals of 10 cc. The overall height of the graduate shall not be less than 9¾ in. (24.8 cm.), nor more than 10¼ in. (26.0 cm.). The graduations shall not be in error by more than 1 cc. at any point on the scale. **Graduate.**

II. PROCEDURE

8. *Sampling.*—Samples should be collected in a previously cooled bottle, preferably by immersing the bottle in the liquid, where possible, and discarding the first sample. Where immersion is not possible the sample should be drawn off into a previously cooled bottle in such a manner that agitation is kept at a minimum. Immediately close the bottle with a tight-fitting stopper, and place in an ice bath or refrigerator capable of bringing the gasoline to a temperature of not less than 32° F. (0° C.), nor more than 40° F. (4.45° C.). **Sampling.**

9. (*a*) The condenser bath shall be filled with cracked ice[1] and enough water added to cover the condenser tube. The temperature shall be maintained between 32 and 34° F. (0 and 1.1° C.). Agitation with air greatly aids in maintaining the required temperature range. **Procedure.**

(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft cloth attached to a cord or copper wire may be used for this purpose.

(*c*) One hundred cubic centimeters of the product shall be measured in the 100-cc. graduated cylinder at 32 to 40° F. (0 to 4.45° C.) and transferred directly to the Engler flask. Both the flask and the graduated cylinder shall have been cooled to a temperature of from 32 to 40° F. (0 to 4.45° C.) before use. None of the liquid shall be permitted to flow into the vapor tube.

(*d*) The thermometer provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that

[1] Any other convenient cooling medium may be used.

the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(*e*) The charged flask shall be placed in the $1\frac{1}{4}$-in. (3.175-cm.) opening in the 6 by 6-in. (15.24 by 15.24-cm.) asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.).

(*f*) The graduated receiver used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 in. (2.54 cm.), but not below the 100-cc. mark. The graduate shall be immersed at least up to the 100-cc. mark in a transparent bath maintained between the temperatures of 32 and 34° F. (0 and 1.11° C.) The top of the graduate shall be covered closely during the distillation with a piece of blotting paper, or its equivalent, and cut to fit the condenser tube tightly. A lead washer, or other suitable material, resting on the blotting paper is a convenient accessory as it serves to hold the paper tightly against the top of the graduate and also provides the additional weight necessary to overcome the buoyant effect of the liquid in the cooling bath.

10. When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of the condensate falls from the condenser in not less than two nor more than five minutes. When the first drop falls from the end of the condenser, the receiving cylinder shall be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the first 10 cc. of distillate shall be recovered in not less than three nor more than four minutes. Thereafter, the rate of distillation shall be maintained uniformly at not less than four nor more than 5 cc. per minute. The reading of the distillation thermometer shall be recorded when the level of the distillate reaches each 10-cc. mark on the graduate.

When the liquid residue in the distillation flask is approximately 5 cc., the heat may be increased because of the presence of heavy ends which have relatively high boiling points. However, no further increase of heat shall be applied after this adjustment. The 4 to 5-cc. rate cannot always be maintained from this point to the end of the distillation, but in no case should the period between the point when approximately 5 cc. of the liquid remains in the flask and the end point be more than 5 minutes.

The heating shall be continued until the mercury reaches a maximum and starts to fall consistently. The highest temperature observed on the distillation thermometer shall be recorded as the end point. Usually this point will be reached after the bottom of the flask has become dry. If the bottom of the flask is not dry, the operator should note this on his report.

The total volume of the distillate collected in the receiving graduate shall be recorded as the "recovery."

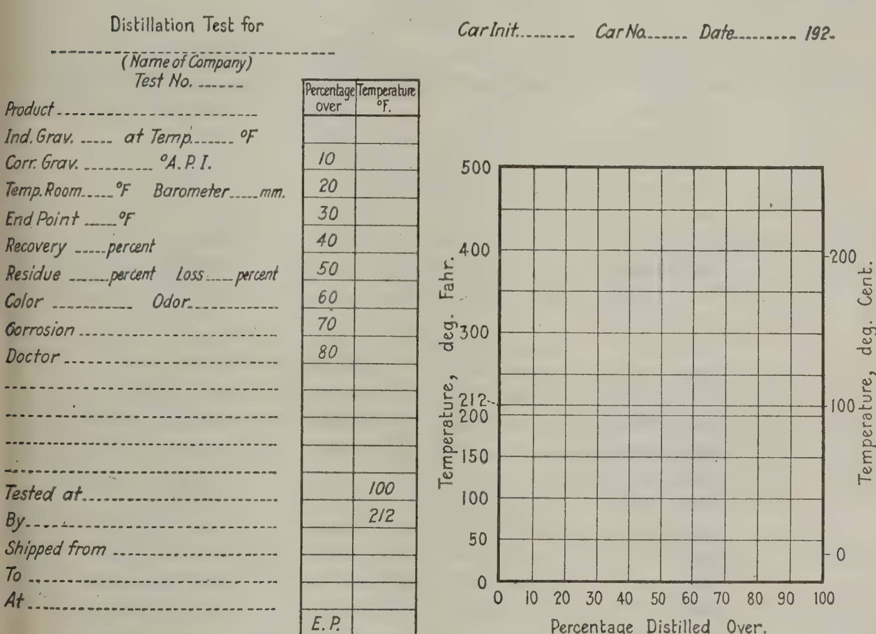

FIG. 3.—Form for Recording Tests on Natural Gas Gasoline.

The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 cc., measured when cool, and the volume recorded as "residue" at 32 to 40° F. (0 to 4.45° C.).

The difference between 100 cc. and the sum of the recovery and the residue shall be calculated and recorded as "distillation loss."

11. *Correction for Barometric Pressure.*—The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute. In such cases the temperature points shall be corrected to 760 mm. (29.92 in.), by the use of the Sydney Young equation, as follows: **Correction.**

For Centigrade readings:

$$C_c = 0.00012\ (760 - P)\ (273 + t_c),$$

For Fahrenheit readings:

$$C_f = 0.00012\ (760 - P)\ (460 + t_f),$$

in which C_c and C_f are, respectively, corrections to be added to the observed temperature t_c or t_f, and P is the actual barometric pressure in millimeters of mercury.

The following table is a convenient approximation of the corrections as calculated by the above equation.

Temperature Range		Correction[1] per 10-mm. Difference in Pressure,	
deg. Cent.	deg. Fahr.	deg. Cent	deg. Fahr.
10 – 30	50 – 86	0.35	0.63
30 – 50	86 – 122	0.38	0.68
50 – 70	122 – 158	0.40	0.72
70 – 90	158 – 194	0.42	0.76
90 – 110	194 – 230	0.45	0.81
110 – 130	230 – 266	0.47	0.85
130 – 150	266 – 302	0.50	0.89
150 – 170	302 – 338	0.52	0.94
170 – 190	338 – 374	0.54	0.98
190 – 210	374 – 410	0.57	1.02
210 – 230	410 – 446	0.59	1.06
230 – 250	446 – 482	0.62	1.11
250 – 270	482 – 518	0.64	1.15
270 – 290	518 – 554	0.66	1.19
290 – 310	554 – 590	0.69	1.24
310 – 330	590 – 626	0.71	1.28
330 – 350	626 – 662	0.74	1.32
350 – 370	662 – 698	0.76	1.37
370 – 390	698 – 734	0.78	1.41
390 – 410	734 – 770	0.81	1.45

Recording Results. 12. The form shown in Fig. 3 is suggested for the recording of results of tests of natural gas gasoline.

[1] To be added in case barometric pressure is below 760 mm., to be subtracted in case barometric pressure is above 760 mm.

TENTATIVE METHOD OF TEST
FOR
MELTING POINT OF PETROLATUM.[1]

Serial Designation: D 127 – 24 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1922; Revised, 1924.

I. DEFINITION.

1. *A.S.T.M. Petrolatum Melting Point.*—The temperature at which petrolatum becomes sufficiently fluid to leave the thermometer used in making the determination of melting point. **Definition.**

II. APPARATUS.

2. (*a*) The melting point thermometer shall conform to the specifications for the A.S.T.M. paraffin wax melting point thermometer, as specified in Section 6 of the Standard Method of Test for Melting Point of Paraffin Wax (Serial Designation: D 87) of the American Society for Testing Materials.[2] **Thermometers.**

(*b*) The bath thermometer shall be of any suitable type accurate throughout the required range to 2° F.

3. The test tubes shall be 25 by 100 mm. in size, and shall be supplied with corks grooved at sides to permit air circulation and bored at the exact center to receive the thermometer. **Test Tubes.**

4. The beakers shall be of the tall form type and have a capacity of 600 cc. **Beakers.**

III. PROCEDURE.

5. (*a*) Samples of sufficient size that exactly represent the petrolatum under inspection shall be secured. The material shall be melted slowly in a casserole or other suitable dish with stirring until the temperature reaches 180° F. The source of heat shall then be removed and the petrolatum allowed to cool to 15° F. above the **Procedure.**

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

[2] 1924 Book of A.S.T.M. Standards.

temperature that it is anticipated will be its melting point. The thermometer bulb shall be chilled to 40° F., wiped dry and while still cold thrust into the melted petrolatum so that approximately the lower half is submerged. It shall be withdrawn immediately, held vertically away from the heat until the surface dulls, and then placed in a water bath having a temperature not over 60° F. for five minutes.

(*b*) The thermometer shall be securely fixed in a test tube by means of a cork so that the lowest point is 15 mm. above the bottom of the test tube. The test tube shall be surrounded with a water bath at a temperature of 60° F. The temperature of the bath shall be raised at a rate of 2° F. per minute to 100° F., then at a rate of 1° F. per minute until the first drop of petrolatum leaves the thermometer. The temperature at this instant shall be recorded.

Melting Point.

6. If the variation of three such determinations does not exceed 2° F., the average of these three determinations shall be reported as the melting point of the sample under test. If the variation of three determinations is greater than 2° F., two additional determinations shall be made and the average of five determinations reported as the melting point.

TENTATIVE METHOD OF ANALYSIS OF GREASE.[1]

Serial Designation: D 128 – 24 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1924.

Scope.

1. These methods of analysis permit determinations sufficiently accurate for referee purposes of all the constituents of greases likely to be covered by specifications. These constituents are fillers and ash, soap bases, soap, fat, water, excess alkali or acid, petroleum products and unsaponifiable matter. No quantitative determination of glycerin is described, but a procedure is outlined for its qualitative detection.

I. SAMPLE.

Sample.

2. The size of sample for the soap determination (Methods (1) and (2) for Determination of Fillers, Soap, Fat, Petroleum Oil and Unsaponifiable Matter) shall be from about 8 to 30 g., depending on the consistency of the grease, which is chiefly determined by the percentage of soap present. Ten to twenty grams is usually a convenient amount for No. 3 Cup Grease, while thin transmission and other greases require a larger sample. The original sample should be stirred or mixed until uniform.

Accuracy of Weighing.

3. Samples need not be weighed more closely than 0.1 g.

II. SOLVENTS.

Petroleum Ether.

4. The petroleum ether shall have an end point not higher than 200° F., and 125 cc. of it shall not consume more than 0.2 cc. of 0.5 *N* potassium hydroxide when a blank test is made as follows:

One hundred and twenty-five cubic centimeters of petroleum ether shall be boiled with 10 cc. of 0.5 *N* alcoholic potassium hydroxide and 50 cc. of neutral 50-per-cent alcohol for $1\frac{1}{2}$ hours on a hot plate, using a glass tube of about 7 mm. internal diameter and 75 cm. long as a reflux condenser. After cooling, the solutions shall be titrated with 0.5 *N* hydrochloric acid and phenolphthalein. Not

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

less than 9.8 cc. of 0.5 *N* acid shall be required for neutralization. The amount of alkali consumed in this test shall be deducted as a blank correction in the fat determination on solution (*E*).[1]

Alcohol. 5. The alcohol shall be prepared from commercial 95-per-cent grain or denatured grain alcohol by distilling from sodium hydroxide, diluting to 50 per cent (or 70 per cent) by volume with distilled water and neutralizing exactly with sodium or potassium hydroxide and phenolphthalein.

Phenolphthalein. 6. The phenolphthalein solution shall be prepared by dissolving 1.0 g. of phenolphthalein in 50 cc. of strong redistilled alcohol, adding 50 cc. of water, and neutralizing with sodium or potassium hydroxide.

Hydrochloric Acid. 7. Concentrated c.p. hydrochloric acid shall be used when "concentrated hydrochloric acid" is specified. Hydrochloric acid, 10-per-cent, shall contain 10 per cent by weight of absolute HCl, with a permissible variation of $\pm$ 0.5 per cent.

III. PROCEDURE.

(*A*) *Determination of Ash.*

NOTE.—The determination of the total ash should not in general be regarded as of any great importance. It is, however, sometimes required. This determination is often unsatisfactory on account of interaction between sodium carbonate derived from the soap, and inorganic fillers. There is always likelihood of reaction with the porcelain crucible itself on account of the long continued heating necessary to burn out all carbon. Moreover, if much sodium or potassium carbonate is present, the ash is fusible and often encloses carbon, making complete removal of the latter very difficult. Results will always be low in the presence of easily reducible oxides of volatile metals. There is also uncertainty as to when calcium carbonate has been completely ignited to calcium oxide. Ash determinations made on the same sample in different laboratories are likely to vary widely. For these reasons, it is usually preferable to make separate determinations of the percentage of fillers and of soap, from which data a calculation is easily made giving all the information to be gained from a direct determination of total ash.

8. The percentage of ash shall not be included in the total of the analysis.

(*1*) *Rapid Routine Method of Ash Determination.*

9. From 2 to 5 g. of grease shall be placed in a weighed porcelain crucible, and the sample weighed to the nearest 0.1 g. If lead or zinc soap is known to be absent, a platinum crucible is more convenient. The combustible matter shall be burned off slowly and the residue finally ignited until the ash is free of carbonaceous matter. The crucible and contents shall then be cooled in a desiccator and weighed, reporting the result as percentage of ash.

[1] Solutions designated by capital letters in parentheses refer to the solutions given in Fig. 1.

(2) Alternative Method of Ash Determination.

10. The sample shall be burned as in method (1) until the ash is nearly free from carbon. The crucible and contents shall then be cooled, the soluble portions dissolved in a little water, and a slight excess of dilute sulfuric acid added, running the acid in carefully from a pipette inserted under a small watch glass covering the crucible. The crucible and contents shall then be warmed on the steam bath until effervescence has ceased. The watch glass shall then be rinsed with water into the crucible. The solution when tested with methyl orange for free acidity shall show free acid present. The contents of the crucible shall be evaporated to dryness, and the whole ignited at a low red heat, adding a few small pieces of dry ammonium carbonate to drive off the excess of sulfur trioxide. After cooling and weighing, the result shall be reported as "percentage of ash as sulfates." **Ash, Alternative.**

The alternative method gives more concordant results than the first method, but requires more time and manipulation.

(B) Qualitative Examination of Ash.

11. An easily fusible ash, dissolving completely in water to give a strongly alkaline solution, indicates a grease containing sodium, potassium, or both. A white infusible ash, practically insoluble in water, but imparting to it an alkaline reaction, may indicate calcium, with or without magnesium or aluminium. Zinc is shown by the yellow color of the ash while hot, and lead may be indicated by the presence of metallic globules, or by the yellow color of the ash when cold. **Detection of Bases.**

12. The ash shall be dissolved in dilute nitric or hydrochloric acid, and the presence of the several bases confirmed by suitable chemical tests, any standard scheme of qualitative analysis being followed.

(C) Quantitative Examination of Ash.

13. For the quantitative examination of ash any standard analytical procedure shall be used, the choice of methods being based on the information gained from the qualitative tests. If only one base is present, a quantitative determination is in general unnecessary.

(D) Determination of Fillers, Soap, Fat, Petroleum Oil and Unsaponifiable Matter.

14. Cup, fiber and sponge greases, with or without graphite, and all comparatively light colored greases, including axle greases, **Choice of Method.**

shall be examined by method (1). Dark greases containing residuum, asphaltic oils and asphalt, tars, etc., shall be examined by method (2), as these ingredients usually cause stubborn emulsions if a shaking-out process is applied at the start.

(1) Determination of Filler, Soap, Fat, Etc.

Determination of Filler.

15. If no fillers are present, Sections 15 and 16 shall be passed over and the grease sample introduced directly from a weighing bottle into a separator, the weight of the sample being obtained by difference. (See Section 2.) The sample shall then be shaken with 75 cc. of petroleum ether and 50 cc. of 10-per-cent hydrochloric acid until the grease is completely decomposed, after which Section 17 shall be followed. If fillers are present, the sample (See Section 2) shall be weighed in a small beaker, 50 cc. of 10-per-cent hydrochloric acid added and the beaker warmed on the steam bath, stirring until all soap lumps have disappeared and the upper layer is clear. If undissolved mineral matter or other filler is present, both layers shall be filtered through a Gooch crucible provided with a suitable mat, the beaker and crucible washed with water and petroleum ether, and the crucible finally washed with strong alcohol, collecting the alcohol washings separately and discarding them. The crucible and contents shall then be dried at 120° C. and weighed, and the result reported as "percentage of insoluble matter" (graphite, mica, talc, asbestos, gypsum, wood pulp, etc.)

Determination of Gypsum.

16. If gypsum is present as a filler, the results obtained in the determination described in Section 15 will be too low because of the solubility of calcium sulfate in hydrochloric acid. A sample of about 5 g. of grease shall be weighed in a small beaker, and decomposed with 50 cc. of petroleum ether and 25 cc. of concentrated hydrochloric acid, warming on the steam bath until all gypsum is dissolved. The contents of the beaker shall be cooled and poured into a separatory funnel, the beaker rinsed with water and a little petroleum ether and the solutions in the funnel allowed to separate clearly. The lower layer shall then be drawn into a beaker, and the petroleum ether in the separator washed with two portions of 20 cc. each of 10-per-cent hydrochloric acid, adding the washes to the strong acid solution in the separator. The hydrochloric acid solution in the beaker shall then be evaporated on the steam bath almost to dryness, diluted to 150 cc. with water, heated to boiling and treated with 10 cc. of 10-per-cent barium chloride solution. The barium sulfate shall then be filtered off and ignited in the usual way, and from the weight of the barium sulfate, the percentage of calcium sulfate shall be calculated, reporting it as $CaSO_4 \cdot 2H_2O$.

NOTE.—Chalk and other forms of alkaline earth carbonates are not determined by this method, but are considered under the determination of free alkalinity, Section 24.

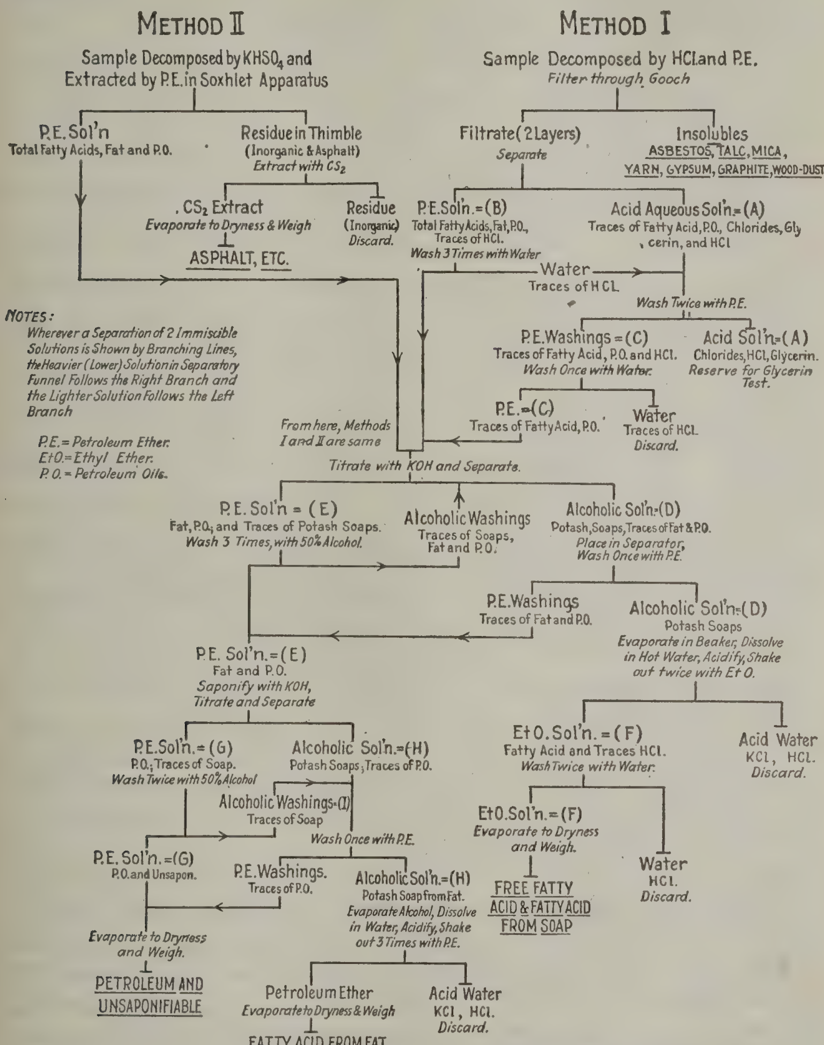

FIG. 1.—Scheme of Grease Analysis.

17. The solution resulting from the decomposition of the filler-free grease or the combined filtrate and washes from the determination of fillers (Section 15), except the alcohol, which is discarded, shall be placed in a separator, allowed to clear, and the aqueous layer (A),[1] **Determination of Soap.**

[1] Solutions designated by capital letters in parentheses refer to the solutions given in Fig. 1.

which contains all the bases as well as glycerin, shall be drawn into another separator. The petroleum ether layer (*B*) shall be washed three times with 25cc. of water to remove hydrochloric acid, adding the washings to (*A*). Solution (*B*) shall then be run into a 250-cc. separator. The aqueous solution (*A*) shall then be washed twice with 20 cc. of petroleum ether (*C*), whereupon (*A*) is set aside for examination for glycerin (see Section 18). Solution (*C*) shall then be washed once with 15 cc. of water, which is rejected, and (*C*) shall be added to (*B*). If (*B*) and (*C*) are comparatively light colored, an approximate determination of free fatty acids and fatty acids from soap may now be made by titrating the solution in the separator with 0.5 *N* alcoholic potassium hydroxide and phenolphthalein, using 200 as the average neutralization value of the fatty acid (that is, 1.0 g. of fatty acid requires 200 mg. of absolute KOH for neutralization). If solutions (*B*) and (*C*) are dark, a few drops of phenolphthalein and sufficient 0.5 *N* alcoholic potassium hydroxide shall be added to make the alcoholic layer distinctly alkaline after vigorous shaking. If the solutions have been titrated, a slight excess of alkali shall be added before separating. The conservative addition of strong alcohol at this point may aid in securing rapid and sharp separation. The two solutions shall be allowed to separate sharply and the lower alcoholic layer (*D*) drawn off into another separator. The upper petroleum ether layer (*E*) shall be washed three times, with 30, 25 and 20 cc. of neutral 50-per-cent alcohol respectively, adding these washes to (*D*). Solution (*D*) shall be washed with 25 cc. of petroleum ether, after which (*D*) is drawn off into a beaker, and the petroleum ether added to (*E*). Solution (*D*) shall then be evaporated to a small volume to remove alcohol, the residue of potassium soap washed into a separator with hot water, acidified with hydrochloric acid and shaken out twice with 50 and 25 cc. of ethyl ether (*F*), respectively. Solution (*F*) shall be run into another separator and washed twice with 20-cc. portions of water, which is discarded. Solution (*F*) shall then be transferred to a weighed beaker and evaporated to dryness on the steam bath, blowing with air to remove all traces of petroleum ether. The residue consisting of free fatty acid and fatty acid from soap shall be heated for a short time on a steam bath, adding and evaporating 5 cc. of absolute alcohol to remove the last traces of water, and weighed. The exact neutralization value shall then be determined on as large a sample of these fatty acids as possible. From the total fatty acid found here, the free acid if any (see Sections 23 and 25) shall be deducted and the

remainder calculated to percentage of soap, using the ash analysis as a guide in distributing the bases, if more than one are present.

The fatty acids may be identified to some extent by special tests, such as odor, crystal form, melting point, iodine number, neutralization value, color reactions, etc.

If the grease is appreciably oxidized, the fatty acids obtained by the ethyl ether extraction are likely to be dark in color and hard to identify. For further study and identification, the neutralized acids may be extracted again (qualitatively) with petroleum ether and dilute HCl, thus eliminating the small amount of more darkly colored matter.

Detection of Glycerin.

18. In case it is desired to determine whether a grease has been made from whole fats or from fatty acids, solution (*A*) shall be neutralized with dry sodium carbonate and sufficient excess added to precipitate calcium or other metals. The whole mass shall then be evaporated to dryness, the residue extracted several times with strong alcohol, the combined alcoholic extracts filtered, and the alcohol evaporated. The residue will then contain most of the glycerin, with a little sodium chloride. The presence or absence of glycerin in the residue shall be confirmed by suitable qualitative tests.

Since fats on saponification yield between 10 and 11 per cent of glycerin, and this analytical scheme does not include a quantitative determination of glycerin, the total percentages in greases made from fats should always be less than 100.

Determination of Fat.

19. The petroleum ether solution (*E*) (Section 17), containing free fat, petroleum oils, and unsaponifiable matter shall be concentrated to a volume of about 125 cc. in a 300-cc. Erlenmeyer flask, 10 cc. of 0.5 *N* alcoholic potassium hydroxide and 50 cc. of strong neutral alcohol added, and the whole boiled on a hot plate with an air condenser for $1\frac{1}{2}$ hours. The uncombined alkali shall be titrated with 0.5 *N* hydrochloric acid, and from the alkali consumed, corrected for the blank determination as specified in Section 4, the percentage of free fat shall be calculated, using 195 as the average saponification value (that is, 1.0 g. of fat requires 195 mg. of absolute KOH for saponification.)

The titrated solution (*G*) shall be placed in a separator, the alcoholic lower layer (*H*) drawn off into another separator and the remaining traces of soap removed as described for (*B*) and (*C*), Section 17, making only two washes with 30 and 20 cc. of 50-per-cent alcohol (*I*). Solutions (*H*) and (*I*) shall be combined, washed once with a little petroleum ether, which is added to solution (*G*), and solutions (*H*) and (*I*) then evaporated to a small volume and the

fatty acid isolated as described for (*D*), Section 17. The percentage of fat may be checked by weighing and titrating the free fatty acid.

Determination of Petroleum Oil.

20. The petroleum ether solution (*G*), now containing all the hydrocarbon oils and unsaponifiable matter shall be evaporated to dryness in a weighed beaker as described for (*F*), Section 17, the residue weighed, and the result reported as "petroleum oils plus unsaponifiable matter."

An approximate determination of the viscosity of the petroleum oil may be made by using a 2 or 5-cc. pipette, which has been standardized against oils of known viscosities. If a complete characterization of the petroleum products is required, a new sample of from 150 to 200 g. of grease shall be decomposed as described above, except that all quantitative operations shall be omitted, as well as the isolation of the free fatty acids, and the use of standard alkali and acid is not necessary.

If the grease contains rosin oil, beeswax, de gras, spermaceti, spermatin, montan wax, candle pitch, and other materials containing a large amount of unsaponifiable substances, the petroleum oils isolated from solution (*G*) will contain the unsaponifiable matter, and the constants found will differ from those of the petroleum products used in making the grease. In most cases, no further separation is possible except in the hands of skillful and experienced operators who can devise special methods to suit the individual conditions.

(2) Determination of Filler, Soap, Fat, Etc.

Method for Dark Greases.

21. The sample shall be weighed in a 3-in. porcelain dish, 10 g. of granulated acid potassium sulfate added and 10 g. of clean dry ignited sand. The dish and contents shall be heated on the steam bath with frequent stirring until all water is driven off, two hours usually being sufficient. After cooling and breaking up lumps with a small pestle, the mixture shall be transferred quantitatively to an extraction thimble, using a little petroleum ether to wash the last traces into the thimble, which should be already placed in a Soxhlet apparatus. The thimble shall be extracted thoroughly with petroleum ether, the extract concentrated somewhat if necessary, and the free fatty acid and fatty acid from soap titrated with 0.5 *N* alcoholic potassium hydroxide as in solutions (*B*) and (*C*), in method (1), Section 17. From this point on, the procedure is identical with that given in method (1), Sections 17, 19 and 20.

Determination of Asphalt and Tarry Matter.

22. The thimble shall be extracted a second time with carbon disulfide, the extract evaporated to dryness, heated for one hour to 120° C. and weighed, the results being reported as asphaltic and tarry matter. The residue in the thimble shall be discarded.

(*E*) *Determination of Free Alkali and Free Acid.*

23. From 10 to 30 g. of grease shall be weighed in a small beaker, dissolved as completely as possible in 75 cc. of petroleum ether by stirring with a spatula, the mixture washed into a 250-cc. Erlenmeyer flask with a small amount of petroleum ether, and the beaker rinsed with 50 cc. of 70-per-cent alcohol, the alcohol being poured into the flask, after which a few drops of phenolphthalein solution shall be added and the whole shaken vigorously. If the alcoholic layer, after settling for a few seconds, is pink, 10 cc. of 0.5 *N* hydrochloric acid shall be added, the solutions boiled on a hot plate for ten minutes to expel carbon dioxide, and the excess acid titrated back with 0.5 *N* alcoholic potassium hydroxide. The free alkalinity shall be calculated in terms of hydroxide of the predominating base. **Free Alkali.**

24. If chalk or any other form of alkaline earth carbonate, or lead carbonate, is present as a filler, it will be detected by effervescence on adding the hydrochloric acid. As the amount of such carbonates is likely to be considerable, the volume of 0.5 *N* hydrochloric acid added shall be increased sufficiently to dissolve all carbonate and leave a slight excess of acid. The solution shall be boiled for two minutes and the excess acid titrated back with 0.5 *N* alcoholic potassium hydroxide, and from the acid consumed, its equivalent in calcium carbonate, etc., shall be calculated, disregarding any other forms of alkalinity which may have been present. **Insoluble Carbonates.**

25. If the original alcoholic layer is not pink, the solution shall be titrated carefully in the cold with 0.5 *N* alcoholic potassium hydroxide, shaking well after each addition. The acidity shall be calculated as oleic acid. **Free Acid.**

26. If soaps of iron, zinc, aluminium, or other weak bases are present, a determination of free acid is not possible, since these metallic soaps react with potassium hydroxide. Up to the present, no means has been devised whereby this determination can be made directly.

(*F*) *Determination of Water.*

27. The quantity of water shall be determined in accordance with the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[1] **Water.**

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE METHOD OF TEST
FOR
PENETRATION OF GREASES[1]

Serial Designation: D 217 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Interpretation of Results. It should be understood that the original consistency of grease is determined by a variety of factors which are very difficult to control. The soap content is the most important single factor, but the kind of fats used, the method of mixing, the final water content, and the temperature of pouring are all very important in determining the final consistency, so that it is impracticable to maintain these consistencies within narrow limits. Any working of the sample after it is poured, or any remelting, will greatly change the consistency. The original consistency also changes to some extent on standing, changing most rapidly during the first 48 hours after manufacture and more slowly thereafter.

Although most tests in the past have been based on the original consistency of greases, this consistency bears no necessary relationship to the worked consistency which is the factor of practical importance for most uses of cup greases, etc. Tests on such greases should therefore be made on the worked consistency. It is also almost impossible to measure the unworked consistency when samples have to be taken from barrels or other large containers.

On the other hand, for very hard greases, such as railroad greases used in slab form, the original or unworked consistency is generally the property of practical importance and should be the object of the test.

Scope. 1. This method shall be used to measure the original consistency or the worked consistency of No. 0 cup grease and all harder greases.

2. The method should be applied only to greases which have not been melted after having been poured and cooled during manufacture.

I. APPARATUS

Penetrometer. 3. The penetration shall be measured on what is customarily known as an asphalt penetrometer such as that described in the Proceedings of the American Society for Testing Materials, Vol. VII, p. 626 (1907), and illustrated in Fig. 3 herein. It is desirable, however, to modify this penetrometer by placing a flat metal plate or

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

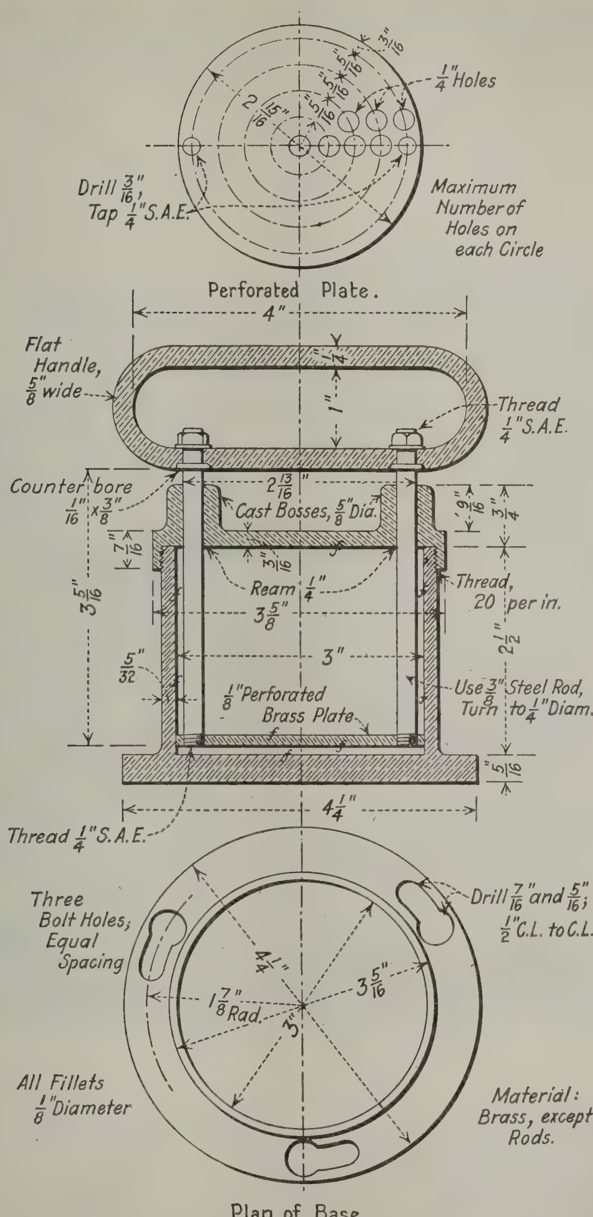

FIG. 1.—Grease Worker.

transite board on top of the present grid base to give a flat support for the grease can. If many tests are to be made, it is preferable to replace the ordinary grid base with a special flat base 6 in. in diameter.

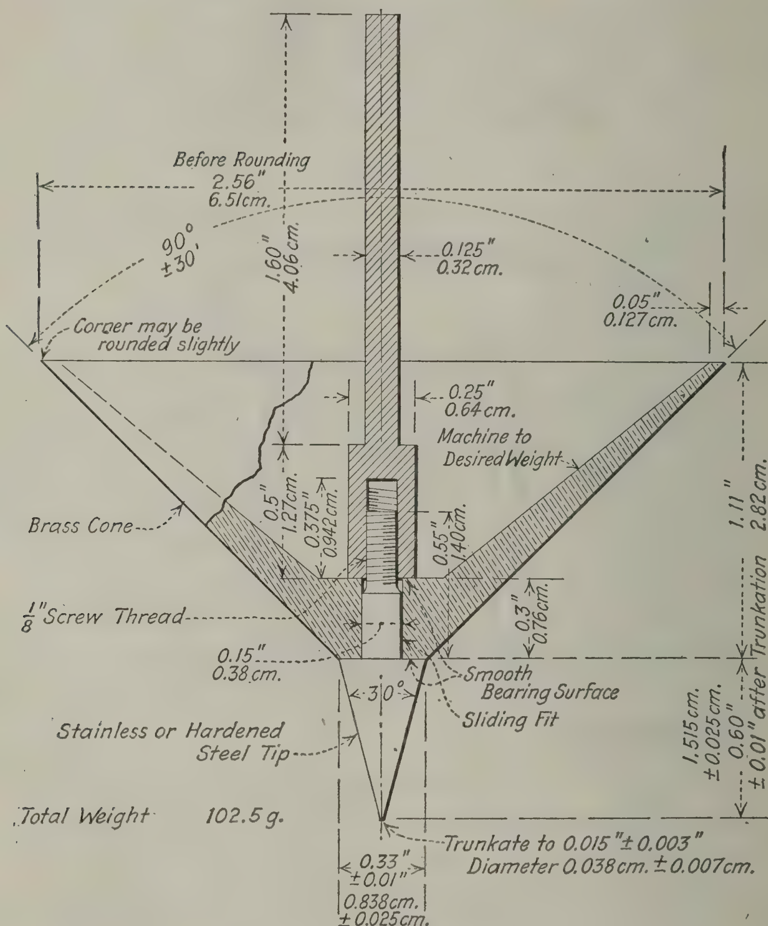

FIG. 2.—Penetrometer Cone.

This plate should have a cork insert in the center to prevent dulling the tip of the cone if it should drop.

Cone and Plunger. 4. The needle used in the asphalt penetration tests shall be replaced with the cone shown in Fig. 2.[1] The cone shall be constructed of stainless steel or of brass with a detachable hardened steel or

[1] The cone may be combined with the asphalt plunger, if desired, providing the total weight of cone and plunger and the outside dimensions of the cone are not thereby changed.

stainless steel tip and made in accordance with the outside dimensions and tolerances shown. The interior construction and dimensions without tolerances are intended to be suggestive only. The outside surface of the cone and tip shall be given a very smooth finish.

5. The total moving weight in the test shall be 150 g. If the asphalt plunger weighs 47.5 g. (50 g. – 2.5 g. needle) the cone or the cone plus an attached weight must weigh 102.5 g. It is in general desirable to make the cone as light as possible so that it **Weight.**

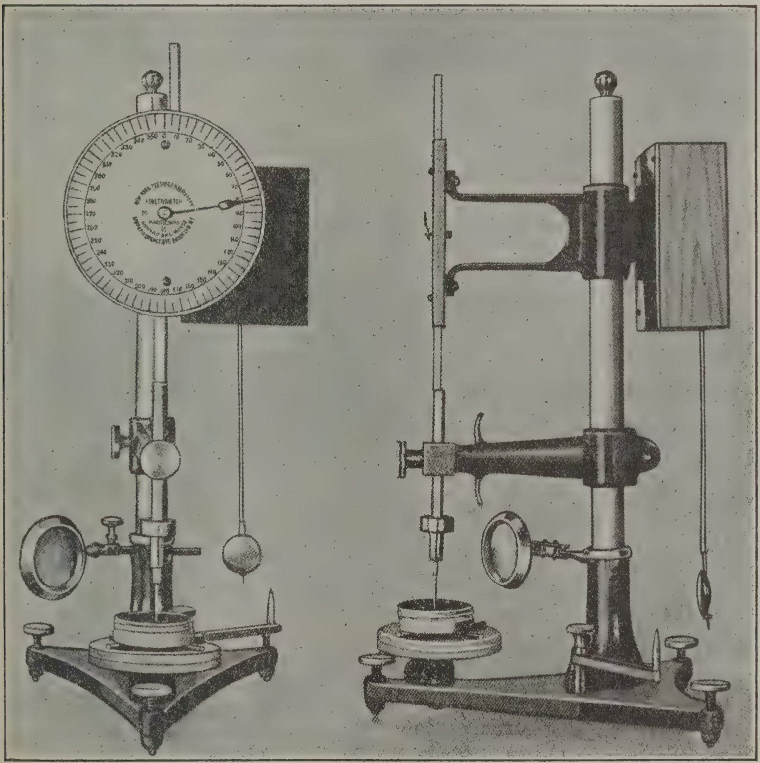

FIG. 3.—Penetrometer.

will have a wide range of usefulness, and to add a weight to give the desired total for the grease test. If the conditions of the tests are not specified, the penetration of a grease shall be understood to refer to the results of this test made at 77° F. (25° C.).

6. The grease worker shown in Fig. 1 shall be used to work the grease to constant consistency in tests where the worked consistency is to be measured. **Grease Worker.**

Constant Temperature Bath. 7. A constant temperature bath regulated to 77° F. ±1° F. (25° C. ±0.5° C.) is desirable to bring the samples to the temperature of test if many tests are to be made.

II. SAMPLES

8. Tests on the unworked consistency of grease shall be made only on grease in the original container (or cake in the case of very hard greases), 1-lb. tins being especially convenient for the purpose. If samples are to be taken from large containers, the tests must be made only on a sample worked as specified below. In taking samples from large containers, any discolored layers near the surface shall first be completely cut away and rejected.

III. PROCEDURE

(A) *Original (Unworked) Consistency*

Bringing to Temperature of Test. 9. The temperature of the sample shall be brought to within 1° F. (0.5° C.) of 77° F. (25° C.) before the test. If the sample is initially within 3 to 4° F. (1.5 to 2° C.) of this temperature, it can be brought to 77° F. (25° C.) by placing in a water bath for 30 or 40 minutes, but if the initial temperature is outside this range it must be placed in the constant temperature bath for $1\frac{1}{2}$ hours to insure reaching the final constant temperature. If the room temperature is more than 3 to 4° F. (1.5 to 2° C.) from 77° F. (25° C.) a lid should be placed on the can, sealing with grease to prevent the entrance of water and the can immersed in the bath for the required period as, otherwise, the temperature of the surface will be different from that of the main body of the grease. Any holes or seams in the can should also be sealed with grease or paraffin.

Preparation of Sample. 10. The surface of the grease shall be cut level and very smooth with a knife. Care must be taken not to work the surface of the grease. Any crust or discolored layers shall be completely removed before testing.

Procedure. 11. The can of grease shall be placed on the penetrometer table and the cone lowered until the tip just touches the surface. Watching the shadow of the tip aids in accurately setting the cone. The scale shall then be set to zero and the plunger released suddenly and kept released for five seconds. The penetration is read from the scale (the units being tenths of a millimeter) by the same procedure as is customary in measuring asphalt penetration as described in the Standard Method of Test for Penetration of Bituminous Materials

(Serial Designation: D 5) of the American Society for Testing Materials.[1]

Spacing of Tests in Cans. 12. In making tests, the total surface area disturbed by the test has a diameter about equal to the measured depth of penetration. In order to prevent one test from being affected by another disturbed area or by sides of the can, in starting a test the tip shall never be placed closer to the sides of the can or the edge of a previous hole than the penetration distance of that particular grease. The grease shall not be smoothed over for further tests.

Number of Tests Required. 13. Five tests shall be made on each sample and the average reported as the consistency, if the mean deviation of these readings does not exceed 3.0 per cent. If the mean deviation does exceed 3.0 per cent, the average of ten readings shall be reported as the consistency of the sample. More than one 1-lb. can will be needed for the required number of tests on the softer greases.

(*B*) *Procedure for Worked Consistency*

Procedure for Worked Consistency. 14. The procedure for worked greases shall be the same as that for unworked consistency except that the grease to be tested shall be transferred to the worker, which shall be filled heaping full with a minimum inclusion of air, brought to within 2° F. (1° C.) of 75° F. (24° C.) and worked with 60 double strokes of the plunger.[2] The top and plunger can then be removed, the surface smoothed over and the test made at once, if the temperature is within 1° F. (0.5° C.) of 77° F. (25° C.). As soon as one test is made, the surface may be smoothed over for the next, taking care to avoid creating air pockets. If desired, the grease may be transferred carefully to another container before bringing to 77° F. (25° C.) and testing. One 1-lb. sample is sufficient for all tests.

[1] A.S.T.M. Standards Adopted in 1925.

[2] In general quite similar results may be obtained by working the grease thoroughly for five minutes with a spatula on a flat plate, providing care is taken to prevent working in much air. For referee tests, however, the standard worker must be used.

TENTATIVE METHODS OF TEST
FOR
VISCOSITY OF PETROLEUM PRODUCTS AND LUBRICANTS[1]

Serial Designation: D 88 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1921; REVISED, 1923, 1924, 1925.

Choice of Instrument. 1. (*a*) Viscosity shall be determined by means of the Saybolt Universal or Saybolt Furol Viscosimeter.

(*b*) In general, the Saybolt Universal Viscosimeter shall be used for lubricants and the Saybolt Furol Viscosimeter for fuel oils and other oils of similar viscosity.

TABLE I.—DIMENSIONS OF OIL TUBES.

Dimensions	Saybolt Universal Viscosimeter			Saybolt Furol Viscosimeter		
	Minimum, cm.	Normal, cm.	Maximum, cm.	Minimum, cm.	Normal, cm.	Maximum, cm.
Inside diameter of outlet tube	0.1750	0.1765	0.1780	0.313	0.315	0.317
Outside diameter of outlet tube at lower end.	0.28	0.30	0.32	0.40	0.43	0.46
Length of outlet tube[1]	1.215	1.225	1.235	1.215	1.225	1.235
Height of overflow rim above bottom of outlet tube[1]	12.40	12.50	12.60	12.40	12.50	12.60
Outside diameter of overflow rim, at the top[1,b]	[a]	....	3.30	[a]	....	3.30
Diameter of container[1]	2.955	2.975	2.995	2.955	2.975	2.995
Depth of cylindrical part of container[1]	8.8	....	....	8.8	....	....
Diameter of container between bottom of cylindrical part of container and top of outlet tube[1]	0.9	....	....	0.9	....	....

[1] This dimension is identical in the Saybolt Universal and the Saybolt Furol instruments.
[a] The minimum value shall preferably be not less than 3.2 cm.
[b] The section of overflow rim shall be bounded by straight lines, except that a fillet is permissible at the junction with the bottom of the gallery.

(*c*) The Saybolt Universal Viscosimeter shall not be used for times of flow less than 32 seconds.

I. APPARATUS

Viscosimeters. 2. (*a*) The Saybolt viscosimeters (see Fig. 1) are made entirely of metal. The oil tube, *A*, is fitted at the top with an overflow cup, *B*, and the tube is surrounded by a bath. At the bottom of the oil tube is a small outlet tube through which the oil to be tested flows into a receiving flask, Fig. 2, whose capacity at 20° C. (68° F.) to a mark on its neck is 60, ±0.15, cc. The outlet tube is of hard and non-

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

corrosive metal such as stainless steel, monel metal, etc. The inside diameter of neck of flask, at the level where the graduation mark is placed, is 0.8 to 1.1 cm. The lower end of the outlet tube is enclosed by a larger tube, which when stoppered by a cork, *C*, acts as a closed air chamber and prevents the flow of oil through the outlet tube until the cork is removed and the test started. A looped string may be

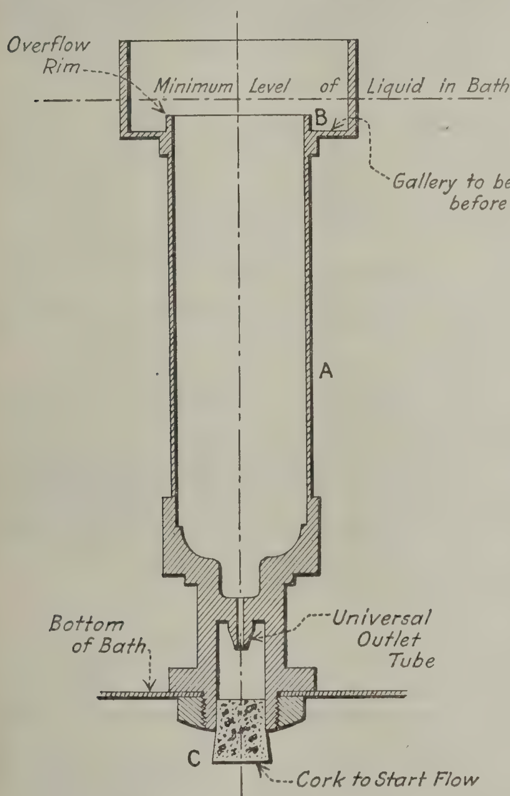

Fig. 1.—Sectional View of Standard Oil Tube.

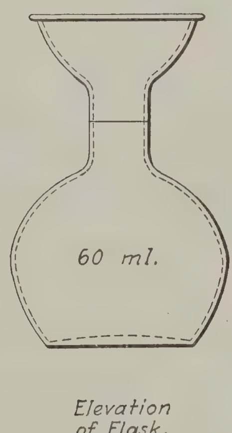

Fig. 2.—Receiving Flask. (This type of flask is recommended as convenient and durable.)

attached to the lower end of the cork as an aid to its rapid removal. The temperatures in the oil tube and in the bath are shown by thermometers. The bath may be heated by any suitable means. The oil tube shall be thoroughly cleaned, and all oil entering the oil tube shall be strained through a 100-mesh wire strainer. A stop watch shall be used for taking the time of flow of the oil and a pipette shall be used for draining the overflow cup.

Oil Tube. (*b*) The oil tubes, which may be standardized by the U. S. Bureau of Standards, shall conform to the dimensions given in Table I. The time of flow shall be within ±1 per cent of the time as obtained with the Bureau of Standards' master tube.[1]

Thermometers. (*c*) The bath and oil tube thermometers shall conform to the following requirements. They cover two sets of 4 thermometers each, one set being graduated in Fahrenheit degrees and the other set in Centigrade degrees, the ranges being chosen to include the temperatures commonly used in testing.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGES AND SUBDIVISIONS:

Range	66– 80° F., subdivision in	0.2° F.	for tests at	77° F.
"	94–108° F., "	0.2° F.	"	100° F.
"	120–134° F., "	0.2° F.	"	122 and 130° F.
"	204–218° F., "	0.2° F.	"	210° F.

or

Range	19–27° C., subdivision in	0.1° C.	for tests at	25° C.
"	34–42° C., "	0.1° C.	"	40° C.
"	49–57° C., "	0.1° C.	"	50° C.
"	95–103° C., "	0.1° C.	"	100° C.

TOTAL LENGTH: 252 to 256 mm. (9.92 to 10.08 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 mm. (0.236 to 0.275 in.). The stem shall preferably be made with an enlargement not less than 4 nor more than 7 mm. (0.16 to 0.28 in.) in length, having a diameter 2 to 3 mm. (0.08 to 0.12 in.) greater than that of the stem, the bottom of the enlargement being 114 mm. (4.5 in.) above the bottom of the bulb.

BULB: Corning normal or equally suitable thermometric glass.
Length, 25 to 35 mm. (1.0 to 1.4 in.).
Diameter, not less than 5 mm. (0.197 in.) and not greater than that of stem.

DISTANCE TO LOWEST SPECIFIED GRADUATION FROM BOTTOM OF BULB: 135 to 150 mm. (5.3 to 5.9 in.).

DISTANCE TO HIGHEST SPECIFIED GRADUATION FROM TOP OF STEM: 20 to 35 mm. (0.8 to 1.4 in.).

CONTRACTION CHAMBER: To be of long narrow type, top to be not more than 60 mm. (2.36 in.) above bottom of bulb, mercury to stand in contraction chamber at 32° F. (0° C.).

EXPANSION CHAMBER: To permit heating the thermometer 90° F. (50° C.) above highest temperature on scale, and in all cases to permit heating to 212° F. (100° C.).

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures and letters clear cut and distinct. The first and each succeeding 1° F. or 0.5° C. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2° F. or 1° C.

IMMERSION: Total.

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[1] This tube represents the average mechanically perfect tube, based on the A.S.T.M. specifications, and has been approved by the American Petroleum Institute Committee on Viscosity Standards.

SPECIAL MARKING: "A.S.T.M." Viscosity test points to be numbered in full figures and in red, other graduations and figures in black. A serial number and the manufacturer's name or trade mark shall be etched on the stem.
SCALE ERROR: The error at any point of the scale shall not exceed 0.2° F. (0.1 C.)
STANDARDIZATION: The thermometers are to be standardized for the condition of total immersion. Correction for emergent stem shall not be applied.
CASE: Each thermometer shall be supplied in a suitable case on which shall appear the marking, "A.S.T.M., Saybolt Viscosimeter Thermometer" and the range.

II. TEMPERATURE OF TESTING

3. (*a*) With the Saybolt Universal viscosimeter, determinations shall be made at 100° F. (37.8° C.), 130° F. (54.4° C.) or 210° F. (98.9° C.). **Temperature.**

(*b*) With the Saybolt Furol viscosimeter, determinations shall be made at 122° F. (50° C.).

(*c*) Viscosities shall be expressed as .... seconds, Saybolt Universal (or Saybolt Furol), being the time in seconds for the delivery of 60 cc. of oil.

(*d*) Fuel oils and other oils of similar viscosity showing a time of less than 25 seconds, Saybolt Furol, at 122° F., shall be tested on the Saybolt Universal at 122° F. Oil showing a time of less than 32 seconds, Saybolt Universal, at 122° F., shall be measured in the Saybolt Universal at 100° F. (37.8° C.). These methods of test do not apply to fuels having a viscosity at 100° F. of less than 32 seconds Saybolt Universal, which are not considered to be fuel oils.

III. PROCEDURE

4. In tests at 100 and 130° F. (37.8 and 54.4° C.), the bath temperature, throughout the test, shall not vary more than ±0.1° F. (0.06° C.) from the predetermined temperature which will maintain thermal equilibrium until the oil tube thermometer is withdrawn. In tests at 210° F. (98.9° C.), a variation of ±0.2° F. (0.12° C.) is permitted. Water shall be used as the bath liquid at 100 and 130° F. (37.8 and 54.4° C.).[1] **Procedure.**

Any construction of bath may be employed provided the bath temperature necessary to maintain thermal equilibrium (while the oil tube is well stirred by the oil tube thermometer) is not in excess of 100.25, 122.35, 130.50 and 212.00° F. (37.9, 50.2, 54.7 and 100.0°C.),

[1] In ordinary routine testing it is frequently desirable to employ oil as a bath medium instead of water. This is allowable provided the temperature of the oil bath is adjusted so that the necessary condition of thermal equilibrium is maintained. It is usually necessary to maintain the oil bath at slightly higher temperatures than are necessary when water is the bath medium. Oil bath temperatures will need to be from 0.1 to 0.2° F. (0.06 to 0.11° C.) higher for tests at 100° F. (37.8° C.) and from 1.5 to 2.0° F. (0.83 to 1.11° C.) higher for tests at 210° F. (98.9° C.) than the corresponding water bath temperatures.

respectively, for the standard temperatures previously mentioned. The level of the bath liquid shall not be lower than 0.5 cm. above the overflow rim of the oil tube. In tests at 210° F. (98.9° C.), the water shall be vigorously stirred, and may be heated by the direct injection of steam near the bottom of the bath. A bath of other suitable liquid may be used provided it is properly heated and stirred.[1] The heating and stirring of the bath for other temperatures may be accomplished by any suitable means, provided the source of heat is not less than the following distances from any part of the oil tube: 2 in. (5 cm.) with an external heater, $1\frac{1}{4}$ in. (3 cm.) with an immersion heater. Viscosity determinations shall be made in a room free from draughts, and from rapid changes in temperature. The room temperature shall be between 68 and 86° F. (20 and 30° C.).[2] All oil introduced into the oil tube either for cleaning or for test shall first be passed through the strainer.

To make the test, heat the oil to the necessary temperature and clean out the oil tube. Pour some of the oil to be tested through the cleaned tube. Insert the cork stopper into the lower end of the air chamber at the bottom of the oil tube, sufficiently to prevent the escape of air, but not to touch the small outlet tube.

Heat the oil to be tested, outside the viscosimeter, to slightly but not more than 3° F. (1.7° C.) above the temperature at which the viscosity is to be determined, and pour it into the oil tube until it ceases to overflow into the overflow cup. By means of the oil-tube thermometer, keep the oil in the oil tube well stirred and also stir well the liquid in the bath. It is extremely important that the temperature of the bath be maintained constant during the entire time consumed in making the test. When the temperature of the bath and of the oil in the oil tube are constant and the oil in the oil tube is at the desired temperature, withdraw the oil tube thermometer; quickly remove the surplus oil from the overflow cup by means of a pipette so that the level of the oil in the overflow cup is below the level of the oil in the tube proper; place the 60-cc. flask, Fig. 2, in position so that the stream of oil from the outlet tube will strike the neck of the flask so as to avoid foam. Snap the cork from its position, and at the same instant start the stop watch. Stir the liquid in the bath during the run and carefully maintain it at the previously determined proper temperature. Stop the watch when the bottom of the meniscus of the oil reaches the mark on the neck of the receiving flask.

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[1] For example, the bath liquid may be water to which glycerin or salt has been added.

[2] These limits are necessary for extreme accuracy in standardization and referee tests, but for routine purposes the use of higher temperatures up to 100° F. (37.8° C.) will not ordinarily cause an error of more than 1 per cent.

The time in seconds for the delivery of 60-cc. of oil is the Saybolt Universal (or Saybolt Furol) viscosity of the oil at the temperature at which the test was made.

With proper attention to details of method of procedure, duplicate results should not differ from each other by more than 1 per cent.

TENTATIVE METHOD OF TEST

FOR

COLOR OF LUBRICATING OILS BY MEANS OF UNION COLORIMETER[1]

Serial Designation: D 155 - 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923

I. APPARATUS

Scope. 1. The color of all lubricating oils shall be determined by means of the Union Colorimeter.

Apparatus. 2. (*a*) The Union Colorimeter, Fig. 1, shall consist of a box, *a*, glass color standards, a glass jar, a light shield, *b*, and a daylight lamp, *c*.

(*b*) The box, *a*, Fig. 1, shall have an interior finish of dull black, and may be constructed of metal or wood, approximately 18 in. in length and $1\frac{1}{2}$ in. in depth in inside dimensions, the observation end being $2\frac{1}{8}$ in. in width and the end next to the source of light 3 in. in width.

A transverse vertical partition having a circular hole in the end $\frac{1}{2}$ in. in diameter shall be placed $1\frac{1}{8}$ in. from the observation end of the box. In the opposite end shall be mounted a rectangular piece of pure white opal glass, approximately $3\frac{1}{4}$ by $1\frac{3}{4}$ by $\frac{1}{16}$ in., which shall be ground or etched on the inner side to diffuse light rays. Approximately $2\frac{1}{8}$ in. from the light end of the box shall be a second vertical partition 1 in. in thickness having two openings about $\frac{3}{4}$ in. in diameter at light-source side and about $\frac{9}{16}$ in. in diameter on the opposite side, symetrically located in regard to the vision line. In case the standards are not mounted, a vertical partition shall be placed approximately $3\frac{1}{4}$ in. from the light-source end, having a thickness of $\frac{1}{8}$ in., and shall have two circular openings $\frac{9}{16}$ in. in diameter and $1\frac{1}{2}$ in. apart. Extending from the opal glass to this partition along the horizontal axis of the box shall be a vertical partition approximately $\frac{1}{8}$ in. in thickness for a distance of $1\frac{5}{8}$ in., and then continuing with a thickness of $\frac{7}{8}$ in. until abutting

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson. Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

against the transverse partition which in conjunction serves to divide the light end of the box into two compartments. In the top of the box, immediately over the centers of these two compartments, there shall be circular holes of such a size to accommodate the standard jar and 4-oz. oil sample bottles. In the top of the box, directly above the $\frac{3}{4}$ in. opening in the left side of the second partition, there shall be an opening or slot suitable for placing the glass color standards in a vertical position

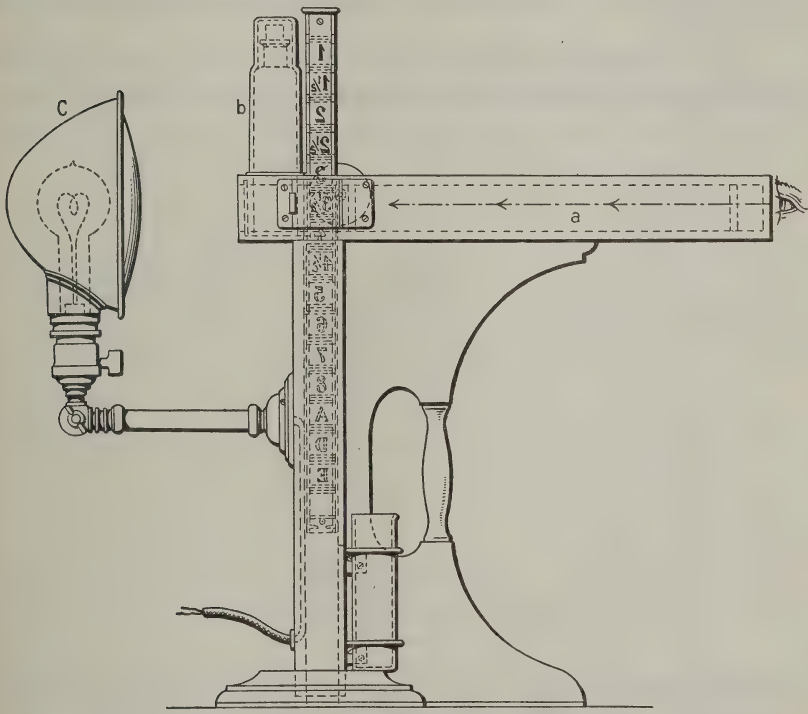

FIG. 1.—Union Colorimeter.

in the opening in the partition. In case the color standards are not mounted in a magazine, this opening shall be provided with a sliding cover so that all light may be excluded. In case all the glass standards are mounted in a magazine, suitable arrangements shall be made for this magazine to be raised or lowered through the opening provided for the glass standards, and when such a magazine is used, a hole may be located in the left side of the box through which the numbers of the mounted glass standards may be read. For convenience, numbers may be stamped on the magazine as mirror images, and a

swinging mirror may be provided on the side of the box so that the operator may read the numbers on the magazine from the front of the instrument.

Color Standards.

(*c*) The glass color standards shall be 15 in number as specified in Table I. These glasses may be kept separately or for convenience may be mounted in a magazine. If so mounted, there shall be a blank opening provided in the magazine so that 2 bottles of oil may be compared in the colorimeter as well as a sample of oil and the glass color standards. The glass color standards shall be not less than $\frac{5}{8}$ in. across.

Glass Test Jar.

(*d*) The standard glass jar, Fig. 2, shall be a true cylinder of clear colorless glass. The internal diameter shall be not less than 32.5 mm.

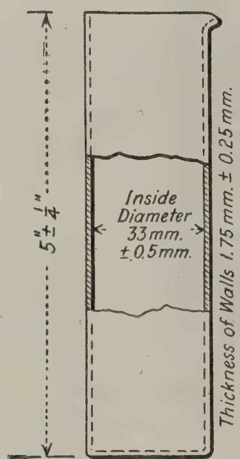

FIG. 2.—Standard Glass Jar for Use With Union Colorimeter.

nor more than 33.5 mm. The thickness of the wall shall be 1.75 mm. ±0.25 mm. The height of the cylinder shall be 5 in. ±$\frac{1}{4}$ in.

Light Shield.

(*e*) The light proof cover, *b*, shall be of any suitable material. It shall be dull black on the inside and of such size as to completely shield all light from the tops of the cylinders, or oil sample bottles, when they are in place in the colorimeter.

Artificial Daylight Lamp.

(*f*) The artificial daylight lamp, *c* (Fig. 1), may for convenience be fastened to the base supporting the box. It shall consist of a 50-watt *C2* national mazda lamp, fitting into a socket on a swivel fitted with a parabolic metal reflector. The inside of this reflector shall be brightly silvered at all times with bright aluminum paint or brightly polished silver or nickel. In the front of the reflector

shall be mounted a dished, but not lenticular, glass color screen of such composition and color as, in conjunction with the *C2* mazda lamp, will yield by spectrum analysis a light closely approximating northern daylight. When in normal position, the color screen shall be 4 in. from the white opal glass.

In case electric current is not available, the colorimeter may be used by exposing the opal glass to northern daylight with no colored objects in the immediate foreground, but the artificial daylight lamp shall be required in referee work.

II. PROCEDURE

3. The oil, when examined, shall be at room temperature unless its cloud point is above the temperature of the room, in which case the **Procedure.**

TABLE I.—GLASS COLOR STANDARDS OF UNION COLORIMETER

A.S.T.M. Color Numbers	Union Petroleum Co. Designations	National Petroleum Association Color Numbers (1915)	National Petroleum Association Names	Lovibond Analysis		
				Red 200 Series	Yellow 510 Series	Blue 1180 Series
1	*G*	No. 1 N.P.A.	Lily White	0.12	2.4	....
$1\frac{1}{2}$	*H*	No. $1\frac{1}{2}$ N.P.A.	Cream White	0.60	8.0	....
2	*I*	No. 2 N.P.A.	Extra Pale	2.5	26.0	....
$2\frac{1}{2}$	*J*	No. $2\frac{1}{2}$ N.P.A.	Ex. Lemon Pale	4.6	27.0	....
3	*K*	No. 3 N.P.A.	Lemon Pale	6.9	32.0	....
$3\frac{1}{2}$	*L*	*a*	Ex. Orange Pale	9.4	45.0	..
4	*M*	No. 4 N.P.A.	Orange Pale	14.0	50.0	0.55
$4\frac{1}{2}$	*N*	No. $4\frac{1}{2}$ N.P.A.	Pale	21.0	56.0	0.55
5	*O*	No. 5 N.P.A.	Light Red	35.0	93.0	....
6	*P*	No. 6 N.P.A.	Dark Red	60.0	60.0	0.55
7	*Q*	*Q*	Claret Red	60.0	106.0	1.8
8	*R*	*b*		166.0	64.0	....
A	*A*	Cylinder Oil Ex. Light Filtered		10.2	29.0	....
D	*D*	Cylinder Oil Light Filtered		21.0	31.0	....
E	*E*	Cylinder Oil Medium Filtered		89.0	56.0	....

[a] N. P. A. Color of 1915 changed slightly to make $3\frac{1}{2}$ more nearly midway between 3 and 4.
[b] A New Color.

oil shall be heated to a temperature of not more than 10° F. above its cloud point. The oil to be examined may be observed in the ordinary 4-oz. sample bottle, but in referee work a standard jar shall be used. The jar shall be placed in the right-hand compartment while in the other compartment shall be placed a 4-oz. sample bottle of distilled water. The jar and bottle shall be covered with the shield, and the artificial daylight lamp lighted. One at a time, the glass color standards shall be placed between the bottle of distilled water and the eye,

in the slot provided for them, until the nearest match possible is obtained. In case the standards are not mounted in a magazine, the slide on the top of the box shall be pressed against the glass standard to keep out light, while making an observation.

Cylinder Oils. 4. Filtered cylinder oils and other oils that are darker than No. 8 color (Table I) shall be diluted with water white kerosene, the color of which is not darker than No. 21 as determined in accordance with the Tentative Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (Serial Designation: D 156 – 23 T) of the American Society for Testing Materials,[1] a mixture of exactly 15 per cent by volume of oil, and 85 per cent by volume of kerosene being used in all cases. This mixture shall then be compared with the three cylinder oil standards, or optionally with the standards ranging from No. 1 to No. 8, inclusive. The daylight lamp may be brought closer to the opal glass in determining the darker colors.

The numbers or letters, designating the color of all oils that have been diluted to make the determination, shall be followed by the designation "Dil."

[1] See p. 374.

TENTATIVE METHOD OF TEST
FOR
COLOR OF PETROLATUM BY MEANS OF THE UNION COLORIMETER[1]

Serial Designation: D 218 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision

Issued, 1925

I. APPARATUS

1. The color of all petrolatums shall be determined by means of the Union Colorimeter. **Scope.**

2. (*a*) The Union Colorimeter, Fig. 1, shall consist of a box, *a*, glass color standards, a glass jar, a light shield, *b*, and a daylight lamp, *c*. **Apparatus.**

(*b*) The box, *a*, Fig. 1, shall have an interior finish of dull black, and may be constructed of metal or wood, approximately 18 in. in length and $1\frac{1}{2}$ in. in depth in inside dimensions, the observation end being $2\frac{1}{8}$ in. in width and the end next to the source of light 3 in. in width.

A transverse vertical partition having a circular hole in the end $\frac{1}{2}$ in. in diameter shall be placed $1\frac{1}{8}$ in. from the observation end of the box. In the opposite end shall be mounted a rectangular piece of pure white opal glass, approximately $3\frac{1}{4}$ by $1\frac{3}{4}$ by $\frac{1}{16}$ in., which shall be ground or etched on the inner side to diffuse light rays. Approximately $2\frac{1}{8}$ in. from the light end of the box shall be a second vertical partition 1 in. in thickness having two openings about $\frac{3}{4}$ in. in diameter at the light-source side and about $\frac{9}{16}$ in. in diameter on the opposite side, symmetrically located in regard to the vision line. In case the standards are not mounted, a vertical partition shall be placed approximately $3\frac{1}{4}$ in. from the light-source end, having a thickness of $\frac{1}{8}$ in., and shall have two circular openings $\frac{9}{16}$ in. in diameter and $1\frac{1}{2}$ in. apart. Extending from the opal glass to this partition along the horizontal axis of the box shall be a vertical partition approximately

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

$\frac{1}{8}$ in. in thickness for a distance of $1\frac{5}{8}$ in., and then continuing with a thickness of $\frac{7}{8}$ in. until abutting against the transverse partition, which in conjunction serves to divide the light end of the box into two compartments. In the top of the box, immediately over the centers of these two compartments, there shall be circular holes of such a size to accommodate the standard jar and 4-oz. oil sample bottles. In the top of the box, directly above the $\frac{3}{4}$-in. opening in the left side of the second partition, there shall be an opening or slot suitable for

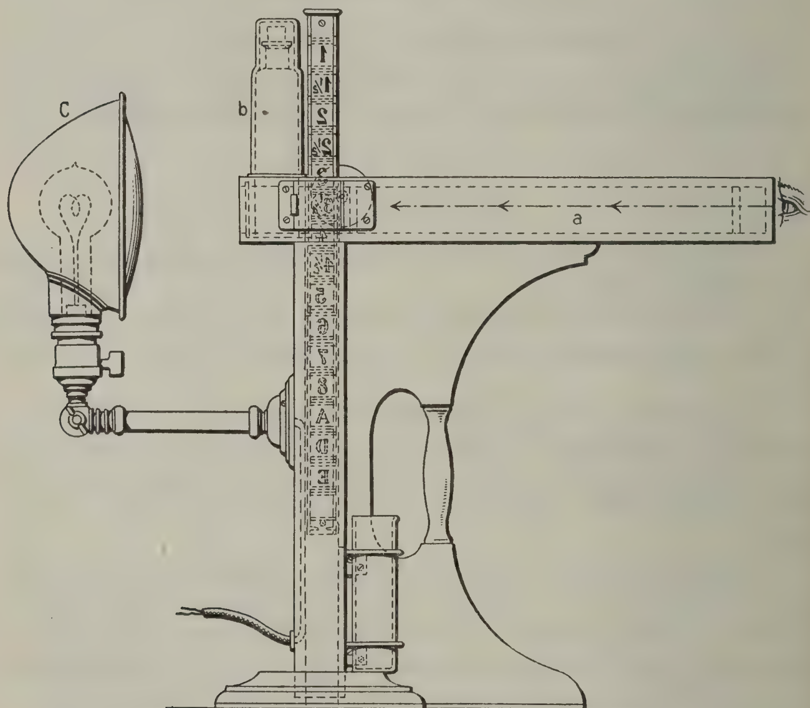

FIG. 1.—Union Colorimeter.

placing the glass color standards in a vertical position in the opening in the partition. In case the color standards are not mounted in a magazine, this opening shall be provided with a sliding cover so that all light may be excluded. In case all the glass standards are mounted in a magazine, suitable arrangements shall be made for this magazine to be raised or lowered through the opening provided for the glass standards, and when such a magazine is used, a hole may be located in the left side of the box through which the numbers of the mounted glass standards may be read. For convenience, numbers may be

stamped on the magazine as mirror images, and a swinging mirror may be provided on the side of the box so that the operator may read the numbers on the magazine from the front of the instrument.

(*c*) The glass color standards shall be 15 in number as specified in Table I. These glasses may be kept separately or for convenience may be mounted in a magazine. If so mounted, there shall be a blank opening provided in the magazine so that a bottle of petrolatum may be compared in the colorimeter with a bottle of oil or with a second bottle of petrolatum as well as a sample of petrolatum and the glass color standards. The glass color standards shall be not less than $\frac{5}{8}$ in. across.

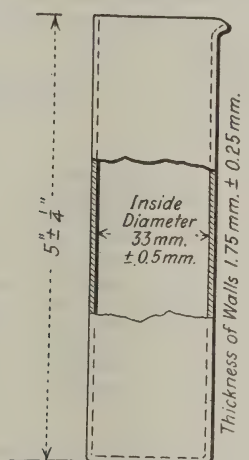

FIG. 2.—Standard Glass Jar.

(*d*) The standard glass jar, Fig. 2, shall be a true cylinder of clear colorless glass. The internal diameter shall be not less than 32.5 mm. nor more than 33.5 mm. The thickness of the wall shall be 1.75 mm. ±0.25 mm. The height of the cylinder shall be 5 in. ±$\frac{1}{4}$ in.

(*e*) The light proof cover, *b*, shall be of any suitable material. It shall be dull black on the inside and of such size as to completely shield all light from the tops of the cylinders, or oil sample bottles, when they are in place in the colorimeter.

(*f*) The artificial daylight lamp, *c* (Fig. 1), may for convenience be fastened to the base supporting the box. It shall consist of a 50-watt *C2* national mazda lamp, fitting into a socket on a swivel fitted with a parabolic metal reflector. The inside of this reflector shall be brightly silvered at all times with bright aluminum paint or brightly polished silver or nickel. In the front of the reflector shall be mounted

a dished, but not lenticular, glass color screen of such composition and color as, in conjunction with the *C2* mazda lamp, will yield, by spectrum analysis, a light closely approximating northern daylight. When in normal position, the color screen shall be 4 in. from the white opal glass.

In case electric current is not available, the colorimeter may be used by exposing the opal glass to northern daylight with no colored objects in the immediate foreground, but the artificial daylight lamp shall be required in referee work.

TABLE I.—GLASS COLOR STANDARDS OF UNION COLORIMETER.

A.S.T.M. Color Numbers	Union Petroleum Co. Designation	National Petroleum Association Color Numbers (1915)	National Petroleum Association Names	Lovibond Analysis		
				Red 200 Series	Yellow 510 Series	Blue 1180 Series
1	G	No. 1 N. P. A.	Lily White	0.12	2.4	....
$1\frac{1}{2}$	H	No. $1\frac{1}{2}$ N. P. A.	Cream White	0.60	8.0	....
2	I	No. 2 N. P. A.	Extra Pale	2.5	26.0	....
$2\frac{1}{2}$	J	No. $2\frac{1}{2}$ N. P. A.	Extra Lemon Pale	4.6	27.0	....
3	K	No. 3 N. P. A.	Lemon Pale	6.9	32.0	....
$3\frac{1}{2}$	L	[a]	Extra Orange Pale	9.4	45.0	....
4	M	No. 4 N. P. A.	Orange Pale	14.0	50.0	0.55
$4\frac{1}{2}$	N	No. $4\frac{1}{2}$ N. P. A.	Pale	21.0	56.0	0.55
5	O	No. 5 N. P. A.	Light Red	35.0	93.0	....
6	P	No. 6 N. P. A.	Dark Red	60.0	60.0	0.55
7	Q	Q	Claret Red	60.0	106.0	1.8
8	R	[b]		166.0	64.0	....
A	A	Cylinder Oil	Extra Light Filtered	10.2	29.0	....
D	D	Cylinder Oil	Light Filtered	21.0	31.0	....
E	E	Cylinder Oil	Medium Filtered	89.0	56.0	....

[a] N. P. A. Color of 1915 changed slightly to make $3\frac{1}{2}$ more nearly midway between 3 and 4.
[b] A new color.

II. PROCEDURE

3. The petrolatum shall be heated to 20 to 30° F. (11 to 17° C.) above the melting point. If darker than No. 8 color (Table I) it shall be diluted with water white kerosine, the color of which is not darker than No. 21 as determined in accordance with the Tentative Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (Serial Designation: D 156 – 23 T) of the American Society for Testing Materials,[1] a mixture of exactly 15 per cent by volume of petrolatum and 85 per cent by volume of kerosine being used in all cases.

[1] See p. 374.

The water white kerosine shall be heated to a temperature 20 to 30° F. (11 to 17° C.) above the melting point of the petrolatum being tested before mixing them together.

This mixture shall then be placed in the standard glass jar and compared with the three cylinder oil standards or optionally with the standards ranging from No. 1 to No. 8, inclusive.

The daylight lamp may be brought closer to the opal glass in determining the darker colors. The numbers or letters designating the color of all petrolatum that has been diluted to make determinations shall be followed by the designation "Dil."

TENTATIVE METHOD OF TEST

FOR

COLOR OF REFINED PETROLEUM OIL BY MEANS OF SAYBOLT CHROMOMETER[1]

Serial Designation: D 156 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923

Scope.

1. The color of all refined oils (naphthas, kerosenes, etc.) shall be determined by means of the Saybolt Chromometer.

I. APPARATUS

Glass Tubes.

2. (*a*) The Saybolt Chromometer shall consist of two glass tubes having an internal diameter of not less than 14 mm. nor more than 16 mm. One glass tube, 20 in. in length, shall be permanently closed at one end with a colorless, plano glass disk, and be mounted in a suitable metal collar, provided with a small pet-cock, for the purpose of draining the tube. This glass tube shall be graduated in $\frac{1}{8}$-in. divisions, numbered in inches. The other tube, 19 in. in length, shall be open at both ends, one end being mounted in a suitable metal collar which will hold the color standards and a black metal diaphragm with a circular aperture 12 mm. in diameter. The mounting of this tube shall be such as to give equal length to both tubes. The tubes shall be mounted vertically and shall be covered at their upper end with a removable diaphragmed metal cap about 1 in. in length and of sufficient diameter to easily slip over the end of the tubes. The circular diaphragm aperture shall be 14 mm. in diameter.

Optical System.

(*b*) The instrument shall be provided with a suitable optical head consisting of prisms and eyepiece. The prisms shall be of a suitable form, matched in their refracting angles and areas, and so mounted as to avoid possibility of disarrangement. They shall be so arranged that the light rays passing through the tubes shall be deflected to a central aperture which is viewed by the eyepiece. The arrangement shall be such as to provide a circular field of vision, the two halves of which are illuminated by the light transmitted by the sample and the color standard, respectively.

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

3. (*a*) Light shall be provided by means of a reflecting mirror with a finely frosted surface set at a suitable angle and so arranged that the reflected light shall pass through suitable openings and through the glass tubes in parallel rays; or it shall be provided by projecting a diffused light directly up through the tubes, from the base of the instrument. **Illumination.**

(*b*) The light shall be supplied by an artificial daylight lamp so arranged as to project a diffused light on the reflecting mirror or directly up through the tubes. **Light Source.**

Table I.—Color Standards, Corresponding to Heights of Oil

Number of Disks	Saybolt Chromometer Height of Oil, in.	Saybolt Chromometer Color Shade	Number of Disks	Saybolt Chromometer Height of Oil, in.	Saybolt Chromometer Color Shade
One........	20.0	+25	Two........	5.50	+4
One........	18.0	+24	Two........	5.25	+3
One........	16.0	+23	Two........	5.00	+2
One........	14.0	+22	Two........	4.75	+1
One........	12.0	+21	Two........	4.50	0
One........	10.75	+20	Two........	4.25	−1
One........	9.50	+19	Two........	4.00	−2
One........	8.25	+18	Two........	3.75	−3
One........	7.25	+17	Two........	3.625	−4
One........	6.25	+16	Two........	3.50	−5
Two........	10.50	+15	Two........	3.375	−6
Two........	9.75	+14	Two........	3.25	−7
Two........	9.00	+13	Two........	3.125	−8
Two........	8.25	+12	Two........	3.00	−9
Two........	7.75	+11	Two........	2.875	−10
Two........	7.25	+10	Two........	2.75	−11
Two........	6.75	+9	Two........	2.625	−12
Two........	6.50	+8	Two........	2.50	−13
Two........	6.25	+7	Two........	2.375	−14
Two........	6.00	+6	Two........	2.25	−15
Two........	5.75	+5	Two........	2.125	−16

(*c*) The artificial daylight lamp shall consist of a *C2* national mazda lamp, the rays of light from which shall pass through a glass color screen of such composition and color, as, in conjunction with the *C2* national mazda lamp, will yield by spectrum analysis a light closely approximating northern daylight. **Daylight Lamp.**

In case electric current is not available, the chromometer may be used by exposing the mirror to northern daylight with no colored objects in the immediate foreground, but the artificial daylight lamp shall be required in referee work.

4. The color standards shall consist of suitable glass disks.

II. PROCEDURE

Illumination.

5. (*a*) The Saybolt Chromometer shall be assembled and the light source so adjusted that a diffused light, with absence of glare or shadow, shall be projected on the reflecting mirror, or projected directly through the tubes from the base of the instrument, thereby eliminating the mirror. Light from all other sources shall be excluded.

Filling Oil Tube.

(*b*) The oil tube shall be cleaned by rinsing with some of the oil to be tested, care being taken to allow the tube to drain thoroughly. The pet-cock on the oil tube shall then be closed and the tube shall be filled with the oil to be tested to a height of 10.5 in.

Determination of Color.

(*c*) The measurement of the color shall be made by using two standard color disks when the oil is darker than the two disks and by using one disk when the oil is lighter than the two disks. After determining the number of disks to be used and with the proper number in place in the color standard, the oil tube shall contain a sufficient height of oil for the color of the oil to be decidedly darker than the color standard, additional oil being added if necessary. The oil shall then be drawn off slowly by means of the pet-cock until the oil appears slightly darker than the color standard. The oil shall then be drawn down to the nearest height corresponding to a standard color shade as shown in Table I. If the color of the oil observed through the eyepiece is still darker than the color standard, the oil shall be drawn down to the next height given in Table I and examined again. This operation shall be continued until the oil and color standard match or show questionable differences. The column of oil shall be lowered one shade more and if the oil is unmistakably lighter than the color standard, the previous color shade shall be recorded as the Saybolt Chromometer Color.

APPENDIX

The following examples of the procedure are given:

Using 1 Disk

Oil darker at height of	16 in.
Oil darker at height of	14 in.
Oil questionable at height of	12 in.
Oil lighter at height of	10.75 in.
Color is	+21

Using 2 Disks

Oil darker at height of	4.5 in.
Oil darker at height of	4.25 in.
Oil questionable at height of	4.0 in.
Oil lighter at height of	3.75 in.
Color is	−2

TENTATIVE METHOD OF TEST
FOR
BURNING QUALITY OF KEROSINE OILS[1]

Serial Designation: D 187 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

1. The burning test for kerosine oil is intended for the determination of the burning quality of ordinary kerosine used for illuminating purposes. **Scope.**

I. APPARATUS[2]

2. *Standard Brass Saybolt Test Lamp.* (See Fig. 1).—The lamp shall conform to the following requirements: **Lamp.**

	INCHES	CENTIMETERS	TOLERANCES, CM.
Inside diameter of burner opening	1.21	3.08	±0.01
Top of fount to top of burner opening	0.45	1.14	0.02
Outside diameter of fount	5.50	13.97	....
Outside depth of fount	2.49	6.33	....
Bottom of fount to bottom of base	5.50	13.97	....
Filling opening			
(1) inside diameter	0.69	1.75	....
(2) distance from side of fount	0.08	0.20	....
(3) height above fount	0.33	0.83	....
Width of rim at bottom of fount	0.65	1.65	....
Thickness of metal	0.03	0.07	....

3. *Miller No. 2 Sun Hinge Burner* (See Fig. 2) or any other burner meeting the following: **Burner.**

The burner shall conform to the following requirements:

	INCHES	CENTIMETERS	TOLERANCES, CM.
Thickness of metal 24 B. & S. gage	....	....	....
Height of cone from base of cone (outside)	1.20	3.05	0.05
Inside diameter of cone at base	1.81	4.60	0.02

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

[2] The equipment used in this test is selected to secure results in a reasonably short time, and it should be understood that this does not indicate any preference as to the proper type for general household use.

	INCHES	CENTIMETERS	TOLERANCES CM.
Inside diameter of cone at base of flame spreader			
(1) parallel to slot	1.43	3.64	0.02
(2) perpendicular to slot	1.43	3.62	0.02
Height of cone from base to a point 1 cm.			
(1) right of center	1.15	2.92	0.06
(2) left of center	1.15	2.92	0.06
Overall diameter of flame spreader at base	2.76	7.01	0.01
Height of collar at flame spreader base	0.31	0.79	0.07
Diameter of scallops	0 21	0.54	....
Depth of scallops	0.08	0.20	....
Center of screw to bottom of collar (outside)	0.29	0.73	....
Holes at base of spreader 28 in number,			
(1) diameter	0.09	0.24	0.01
(2) spacing, edge to edge	0.14	0.35	0.02
Space at back of flame spreader collar (without holes)	0.83	2.10	....
Space at front of flame spreader collar (without holes)	0.61	1.55	....
Width of flame spreader slot at top			
(1) center	0.39	1.00	0.03
(2) 1 cm. to left of center	0.39	1.00	0.03
(3) 1 cm. to right of center	0.39	1.00	0.03
Width of wick holder (outside)	0.96	2.44	0.01
Depth of wick holder (outside)	0.17	0.44	0.01
Length of wick holder	1.75	4.45	0.02
Width of air vent (outside)	0.34	0.87	....
Depth of air vent (outside)	0.08	0.20	....
Length of air vent	1.67	4.25	0.01
Top of wick holder to base of flame spreader slot	0.03	0.08	0.06
Base of flame spreader slot (inside) to edge of wick holder (outside)	0.23	0.59	0 07
Edge of flame spreader slot (top center) to top center edge of wick holder	0.11	0.29	0.05

Wick holder (to run parallel to flame spreader slot.)

LOWER HALF:

Diameter at widest point (parallel to slot)	2.22	5.64	0.02
Diameter at first constriction	2.18	5.53	0.02
Diameter at second constriction	1.96	4.97	0.02
Diameter at third constriction	1.42	3.60	0.02
Diameter at fourth constriction	1.22	3.10	0.02
Diameter at bottom	1.19	3 02	0.02
Ventilating openings, 12 in number, width,			
(1) center	0.29	0.73	0.01
(2) top and bottom	0.21	0.54	0.01
Ventilating openings, height,			
(1) center	0.24	0.61	0.01
(2) sides	0.32	0.81	0.01
Top of wick holder to top of holder support	1.01	2.57	0.03
Top of lower half of burner to outside holder support	0.62	1.57	0.02

SCREEN:	INCHES	CENTIMETERS	TOLERANCES, CM.
Diameter of screen	2.12	5.38	0.02
Length of slot in screen	1.01	2.56	0.05
Width of slot (1) narrowest point	0.20	0.52	0.02
Width of slot (2) widest point	0.30	0.75	0.02
Diameter of holes in screen	0.05	0.12	0.03
Number of holes in screen per sq. cm., 45, with a tolerance of 7			
Height of screen from base to top (over-all)	0.11	0.29	0.01
Diameter of screen at top	1.16	2.94	0.02
Diameter of screen at rim	0.10	0.26	0.01

NOTE.—The burner shall be used as received from the manufacturer provided it meets the above specifications and is free from gross imperfections of manufacture. It shall be clean and in good condition at the start of the test, but emery paper shall not be used in cleaning nor imperfections removed by means of a file or otherwise.

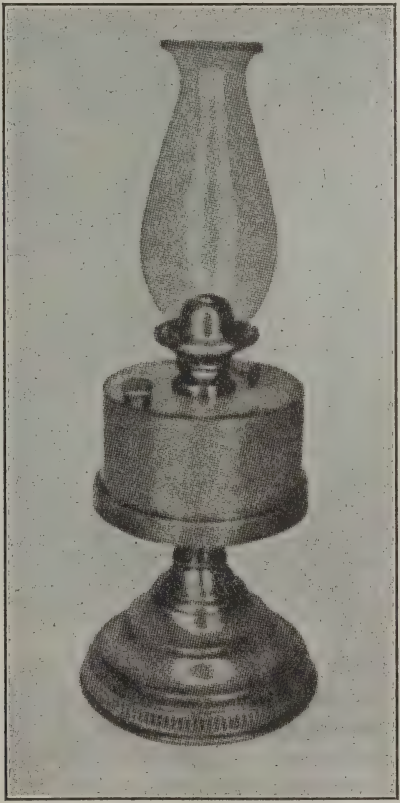

FIG 1.—Test Lamp and Chimney.

4. *Macbeth-Evans No. 514 Pearl Top Chimney.* (See Fig. 1.)— **Chimney.** The chimney shall conform to the following requirements, all figures being outside measurements:

	Inches	Centimeters	Tolerances, cm.
Total height	8.06	20.48	0.50
Diameter of constriction			
(1) upper	1.77	4.50	0.08
(2) lower	2.27	5.77	0.07
Diameter at widest point	3.69	9.36	....
Diameter at top	2.83	7.20	0.09
Diameter at bottom	2.60	6.61	0.06
Distance from bottom to narrowest point	7.03	17.86	0.55
Distance from bottom to widest point	2.47	6.27	0.55
Distance from bottom to constriction near bottom	0.30	0.77	0.19
Thickness of glass at constriction			
(1) top	0.07	0.19	0.05
(2) bottom	0.05	0.12	0.03

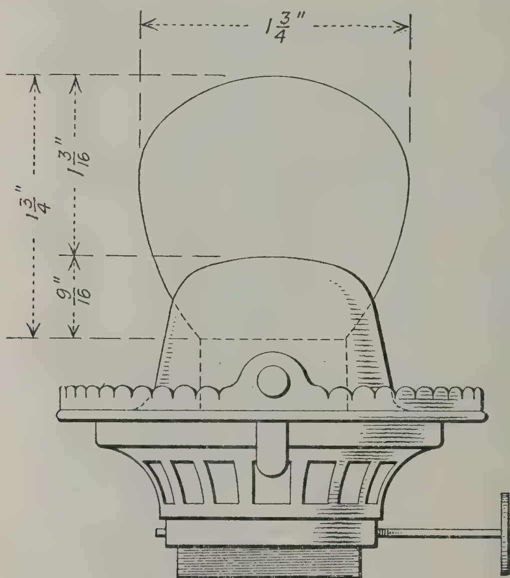

FIG. 2.—Burner.

Wick. 5. *American Wick Co.'s "B" or Similar No. 2 Wick.*—The wick shall be used as received from the manufacturer and shall not be extracted with ether or other solvent previous to use. A wick shall be rejected if it fits the wick guide too loosely or too tightly, and a new wick shall be used for each burning test.

Sight Gage. 6. A sight gage for measuring the flame height and width accurate to $\frac{1}{16}$ in. shall be used. (See Fig. 3.)

Room. 7. A special room for conducting the burning test is desirable but the lamp may be burned in any part of any adequately ventilated room

reasonably free from drafts. The temperature of the room is immaterial, but is shall not vary more than 20° F. during the test.

II. PROCEDURE

8. The lamp shall be cleaned and 850 cc. of the oil to be tested shall be poured into the fount. The wick, wetted with the oil, shall be inserted in the burner, and the burner shall be firmly screwed into the **Setting the Lamp.**

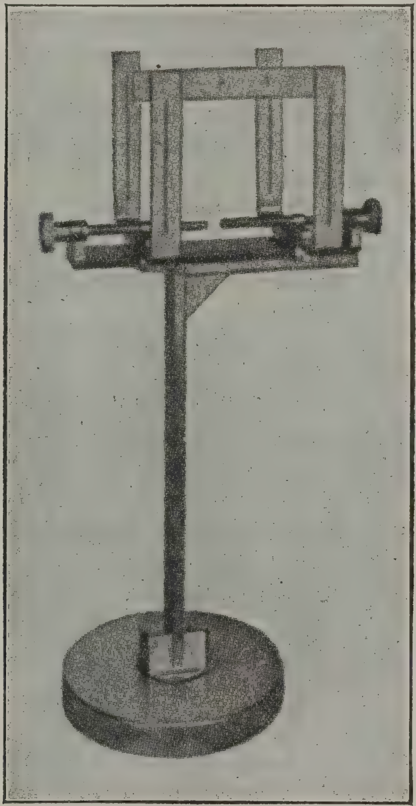

FIG. 3.—Sight Gage.

fount. The wick shall be trimmed with the scissors so as to produce a flame, which, unless otherwise specified, shall be $1\frac{3}{4}$ in. in height measured from the top of the wick guide and $1\frac{3}{4}$ in. in width at its widest point. (See Fig. 2.) The trimming should be done very carefully to produce a smooth symmetrical flame free from peaks or ears, and the lamp should be lit and the flame inspected from time to time during the course of trimming in order to determine whether the trim is satisfactory. Proper height can be obtained by turning the

wick up or down and the proper width obtained by trimming the corners of the wick. These adjustments should be made to $\pm\frac{1}{16}$ in. from the standard flame dimensions. The lamp shall be allowed to burn for one hour, and the flame then readjusted, if necessary, to the standard dimensions. At the end of the first hour the lamp shall be weighed, while burning, on a platform balance accurate to 1 g., and weighed again after exactly 60 min. further burning. The oil consumed shall be calculated in cubic centimeters per hour. The initial rate of oil consumption, thus established, shall not vary from 43 cc. per hour by more than ± 2 cc. With proper attention to the flame dimensions this measurement is needed only as a check, since with a given flame size and shape the initial rate of oil consumption will always be within the limit set.

Conducting the Test. 9. The oil shall be allowed to burn continuously, without further adjustment of any kind, for the duration of the test. The lamp shall be refilled to within $\frac{1}{4}$ in. of the top of the fount, at intervals of 8 hours, without putting out the flame.

Duration of the Test. 10. Unless otherwise specified, the duration of the test shall be 24 hours of continuous burning.

Interpretation. 11. At the end of the test, the height and width of the flame and the condition of the chimney, flame, and wick, shall be noted. The final flame measurement shall be taken without refilling the lamp.

Reproducibility. 12. With care and proper attention to detail, the average oil consumption per hour should not differ in duplicate determinations by more than 2 cc. With this condition fixed, the other characteristics of the oil will be reproduced invariably, with the exception of initial mushroom formations ("ears," "niggers," "toadstools"), reproducibility of which will vary with the quality of the oil, amounting in the poorer oils to 10 per cent of the burning time.

TENTATIVE METHOD OF TEST
FOR
BURNING QUALITY OF LONG-TIME BURNING OIL FOR RAILWAY USE[1]

Serial Designation: D 219 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

1. The burning test for long-time burning oil is intended for the determination of the burning quality of special kerosine oil used in railway semaphore signal lamps. **Scope.**

I. Apparatus

2. The lamp shall be the standard American Railway Association, Signal Section, semaphore lamp. **Lamp.**

3. A 31 fluid ounce fount, about 3 in. high, measured from inside the bottom to top of burner collar, and fitting the standard lamp, shall be used. **Fount.**

4. The burner shall be a *Dressel No. 23 Long-Time Burner*, New York Central Type, or any other burner meeting the following requirements: A nickel plated long-time burner made of suitable weight metal, provided with the conventional wick adjusting device, with any convenient means of fitting to fount, and in other respects corresponding to the following requirements: **Burner.**

Flame Spreader.—Cylindrical, $\frac{5}{16}$ in. in height and $\frac{1}{2}$ in. in inside diameter, soldered to the top of wick tube. Two V-shaped slots, placed opposite to each other, with top of V $\frac{1}{4}$ in. wide and bottom ending just above top of wick tube.

Wick Tube.—$1\frac{15}{16}$ in. in length, $\frac{1}{4}$ in. in inside diameter, and extending $\frac{5}{8}$ in. above top of wick tube.

Chimney Support Ring.—$1\frac{1}{4}$ in. in inside diameter, $\frac{7}{16}$ in. below top of flame spreader, equipped with 4 springs to hold chimney in place.

Air Vents.—Four $\frac{1}{16}$ in. holes placed symmetrically in flame spreader just above the top of the wick tube; four rectangular

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

openings, $\frac{7}{16}$ in. high, by $\frac{9}{16}$ in. wide, placed below the chimney support ring and between the chimney springs.

Chimney. 5. The chimney shall be a Macbeth No. 55 D Chimney, or any other chimney meeting the following requirements: Straight wall glass $2\frac{1}{2}$ in. high and $\frac{1}{8}$ in. in inside diameter, with rim at top and bottom.

Wick. 6. A new gray round felt wick, approximately $7\frac{3}{4}$ in. long and $\frac{3}{16}$ in. in diameter, made by sewing a strip of felt $\frac{1}{2}$ in. wide by $7\frac{3}{4}$ in. long by $\frac{1}{8}$ in. thick into proper form is suitable. The wick shall be washed with redistilled ether and dried at room temperature before use.

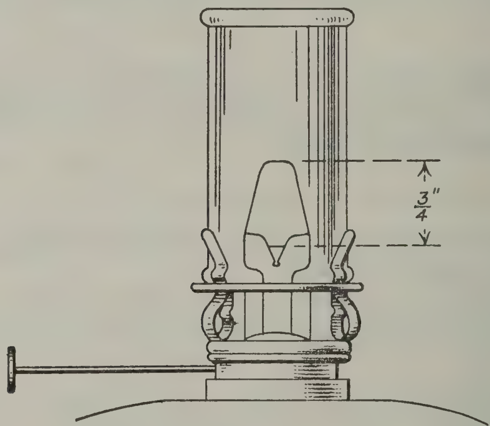

Fig. 1.

Balance. 7. A platform balance sensitive to 1 g. shall be used.

Room. 8. The lamp may be burned in any well ventilated room, or out of doors, if not subjected to the direct rays of the sun.

Sight Gage. 9. A sight gage, or other suitable flame measuring device, accurate to $\frac{1}{16}$ in. shall be used. The sight gage shown in Fig. 2 is satisfactory.

II. Procedure

Procedure. 10. Seven hundred and fifty cubic centimeters of the oil to be tested shall be placed in the fount. The burner, wick, chimney and fount shall be assembled, the rough edge trimmed from the wick and the wick centered in the flame spreader, lighted and adjusted to give a flame $\frac{3}{4}$ in. high, measured from top of wick. This, and all following flame measurements, should be made with the assembly inside of the lamp, with slide open, and in a room free from drafts. The flame should have a flat top and be of the general shape indicated

in Fig. 1. If this cannot be accomplished by leveling the lamp, the burner should be rejected. Inability to secure the specified flame shape will be due, in such cases, to improper alignment of the sides of the flame spreader. It is essential that the specified flame shape be adhered to, as any deviation from it may influence the rate of oil consumption and the flame drop. The assembly shall be weighed

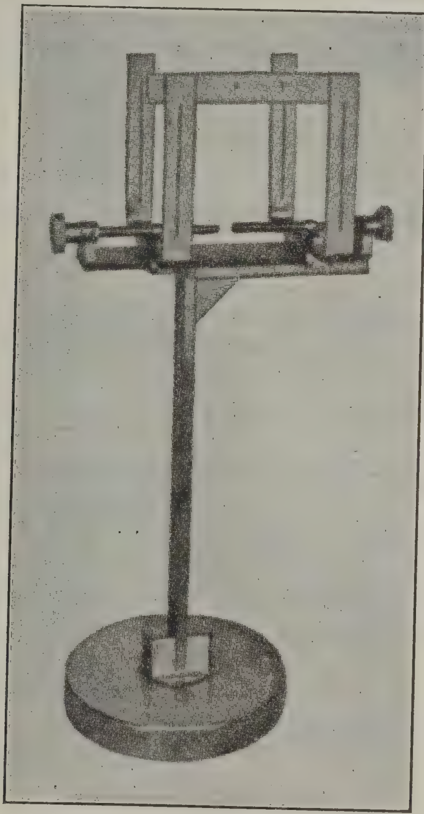

FIG. 2.—Sight Gage.

while burning, then placed in the lamp. The wick shall be readjusted, if necessary, without removing the fount from the lamp, during the first four hours. The lamp shall be allowed to burn continuously for the duration of the test. After 96 hours, the fount assembly shall be removed from the lamp, weighed while burning and then replaced in the lamp. After 120 hours the lamp shall again be weighed and the average rate of oil consumption per hour calculated for the 96 to

120-hour period. From this rate and the original weight of oil in the fount, the number of additional hours required to make the total quantity of oil consumed 650 cc. shall be calculated and the lamp burned the calculated length of time. The oil consumption shall be verified by weighing at the end of the test. The time required to burn 650 cc. will, in general, be not less than 120 hours and not more than 144 hours if proper attention is paid to initial flame height and shape. At the end of the test, observe and record the change in height of flame, and condition of the wick and chimney.

TENTATIVE METHOD OF TEST
FOR
SULFUR IN NAPHTHAS AND ILLUMINATING OILS.[1]

Serial Designation: D 90 – 24 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1924.

This method is intended to be applied to illuminating oils and petroleum naphthas. It is not applicable to mixtures containing carbon bisulfide. **Scope.**

APPARATUS.

Absorber of chemically resistant glass, about 150-cc. capacity, containing glass beads or short pieces of glass rod in the suction side as shown.

Chimney of chemically resistant glass connected with the absorber by a rubber stopper.

Spray trap of chemically resistant glass connected with the absorber by a rubber stopper.

Small lamp of about 25-cc. capacity. This lamp may conveniently consist of a 25 to 35-cc. Erlenmeyer flask and a cork carrying a short section of glass tubing, about $\frac{1}{8}$ in. in inside diameter. The cork must be grooved along the sides so that air may enter the flask while the oil is being consumed.

Ordinary cotton wicking.

Filter pump or other means for continuous suction and rubber tubing to connect with spray trap.

SOLUTIONS REQUIRED.

Hydrochloric acid.—Solution containing 2.275 g. HCl per liter and carefully checked for accuracy.

Sodium carbonate.—Solution containing 3.306 g. Na_2CO_3 per liter. Exactly 10.0 cc. should be required to neutralize 10.0 cc. of the hydrochloric acid solution.

Methyl orange.—Solution in distilled water, containing 0.004 g. methyl orange per liter.

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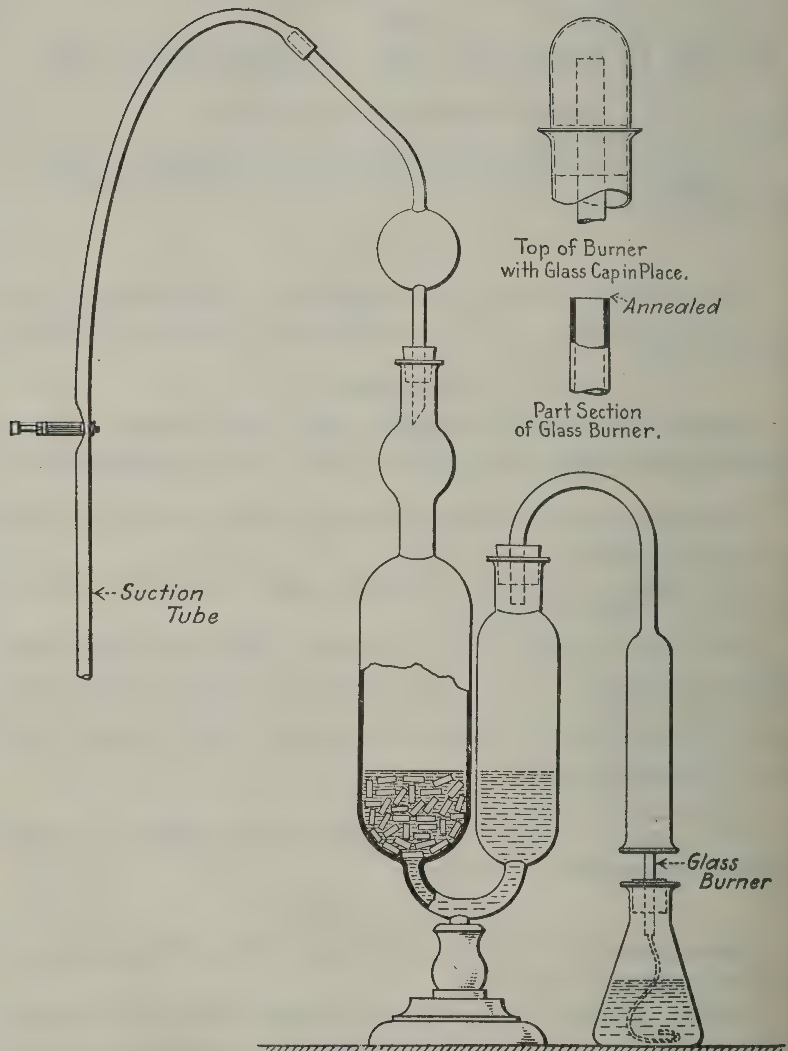

FIG. 1.—Apparatus for Determination of Sulfur in Oils.

PROCEDURE.

Pass two strands of new cotton wicking about 4.5 in. long through the $\frac{1}{8}$-in. diameter wick tube so that they are not twisted but parallel in the wick tube. Trim the wick with very sharp scissors. Pour into the clean dry lamp about 20 cc. of the oil to be tested, insert the wick and cork and weigh the assembly with an accuracy of 0.001 g. It is advisable to make a blank determination at the same time and under the same conditions by burning sulfur-free alcohol in a similar lamp.

Rinse out the absorber containing the glass beads thoroughly with distilled water and add exactly 10.0 cc. of the standard sodium carbonate solution from an accurately calibrated burette, allowing the burette to drain for three minutes before taking the reading. Rinse the chimney and the spray trap with distilled water, dry the chimney and connect both to the absorber as shown in Fig. 1. Set up the apparatus for the blank determination in exactly the same manner and using exactly 10.0 cc. of the sodium carbonate solution. Apply gentle suction to both absorbers, light both the weighed oil lamp and alcohol lamp and then place in position under the chimneys so that the tops of the wick tubes extend into the chimneys not more than $\frac{1}{16}$ in. Adjust the wick height and the suction so that the flame is steady, free from smoke and approximately $\frac{1}{4}$ in. high. This requires that the wick be flush with the top of the wick tube for naphthas and a little higher for illuminating oils. The room must be free from drafts. The suction on the blank should be so adjusted that air is drawn through both determinations at the same rate. Continue burning for about two hours, or less if the sulfur content of the oil is high. During this time the oil should be consumed at the rate of about 1 g. per hour.

Extinguish the flames and stop the suction on both absorbers. Weigh the oil lamp immediately and calculate by difference the weight of oil consumed. Working with the blank first, disconnect the spray trap and chimney and wash them thoroughly with the methyl orange solution, using a wash bottle with a very fine jet and collecting the washings in the absorber. The amount of solution required for washing should not exceed 35 cc. Carefully titrate the very faintly yellowish solution in the absorber with standard HCl, added to the suction side of the absorber from an accurately calibrated burette. During this titration, the contents of the absorber should be agitated carefully, either by blowing through a rubber tube held between the operator's lips and connected at the other end with the chimney side of the absorber or else by the use of a suitable rubber syringe bulb.

As the end point is approached, draw the liquid back into the chimney side between each addition of acid and then blow it into the suction side, agitating as before. As soon as the first permanent pink color appears, the end point has been reached. Read and record the volume of HCl solution used.

Rinse the chimney and spray trap used in the actual determination into the absorber to which they were connected, exactly as prescribed for the blank. If the methyl orange solution in the absorber has a pink color, too much oil has been burned and the determination must be repeated, burning for a shorter time. Titrate just as in the blank, making sure that the absorber is cold. Read and record the volume of HCl solution required.

Calculate the sulfur content of the oil by substituting the proper values in the following formula:

$$\text{Percentage of Sulfur} = \frac{(\text{HCl for blank, cc.} - \text{HCl for sample, cc.}) \times 0.1}{\text{grams of oil burned}}$$

If a blank is not run, the formula is:

$$\text{Percentage of Sulfur} = \frac{(\text{Na}_2\text{CO}_3\text{, cc.} - \text{HCl, cc.}) \times 0.1}{\text{grams of oil burned}}$$

These formulas are correct only for the standard solutions specified, 1 cc. of each being equivalent to 0.001 g. of sulfur. The use of solutions of any other strength, such as N/10, involves more complicated calculation and is not advisable.

TENTATIVE METHOD OF TEST
FOR
SULFUR IN PETROLEUM OILS HEAVIER THAN ILLUMINATING OIL.[1]

Serial Designation: D 129 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922.

I. APPARATUS.

1. The oxygen bomb shall have a capacity of not less than 300 cc., and shall be of a design or construction such that no leaks shall occur at any pressure or temperature generated during the test and such that when open, liquid contents can be easily and completely drained. The inner surfaces shall be of materials that are chemically and physically resistant to the process or products of combustion. The gaskets, insulating materials, etc., shall be, as far as possible, physically and chemically resistant and in no event shall they undergo any reaction which would increase or decrease the sulfur content of the bomb liquors. **Oxygen Bomb.**

2. The oil cup shall be of platinum, glazed silica, or other suitable material, with a capacity of not less than 2.5 cc. nor more than 5.0 cc. **Oil Cup.**

3. If a platinum oil cup is used, the fuse wire shall be of platinum; if a glazed silica oil cup is used, the fuse wire may be of either platinum or iron. No. 35 B. & S. gage is a convenient size. **Fuse Wire.**

4. (*a*) The distilled water and all reagents should be sulfur free, but in such cases where it is necessary to employ reagents not sulfur free, blanks shall be run and the figures thus obtained used to correct the results of actual determinations. **Reagents.**

(*b*) The barium chloride solution shall contain 100 g. of $BaCL_2 \cdot 2H_2O$ per liter.

II. PROCEDURE.

5. (*a*) Twenty cubic centimeters of distilled water shall be placed in the bottom of the bomb. From 0.6 to 0.8 g. of the oil to be tested **Procedure.**

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

shall be placed in the weighed oil cup and the weight of this charge shall be determined to an accuracy of at least ±0.002 g. The cup shall be placed in the proper position in the bomb, the ignition mechanism arranged and the bomb closed. Oxygen shall be admitted slowly until a pressure is reached as indicated by the following table:

Capacity of Bomb, cc.	Minimum Gage Pressure, Atmospheres.
300 to 350	40
350 to 400	35
400 to 450	30
450 to 500	27.5
Above 500	25

The leads from the firing circuit shall be attached, the bomb placed in a bucket of cold water, and ignited. The bomb shall be allowed to stand in the water for 10 minutes and shall then be removed. The valve of the bomb shall be opened, allowing the gas to escape at an approximately even rate so that the pressure is reduced to atmospheric in not less than 1 minute. The bomb shall be opened, and all parts of its interior, including the oil cup, rinsed with a fine jet of distilled water. All washings, which should not amount to more than 350 cc., shall be collected in a beaker. Particular care should be taken not to lose, by splashing, or otherwise, any of the liquid contents of the bomb. The washings shall be filtered through a washed "qualitative" filter paper. The filter shall be washed thoroughly. Two cubic centimeters of concentrated HCl and 10 cc. of saturated bromine water shall be added to the filtrate. The solution shall be evaporated to about 75 cc. on a steam bath or hot plate. Ten cubic centimeters of hot barium chloride solution shall be added in a fine stream or drop-wise to the hot solution, stirring during the addition and for two minutes afterward. The solution shall be allowed to stand over night, or shall be kept hot for one hour on the steam bath or hot plate, allowing the precipitate to settle for another hour, while cooling. The supernatant liquid shall be filtered through an "ashless quantitative" filter paper, the precipitate washed with water, first by decantation, then on the filter, till free from chloride. The paper and precipitate shall be transferred to a suitable weighed crucible, dried at low heat till moisture is evaporated, the paper charred (without flaming), and finally ignited at a good red heat till the precipitate is just burned white. A satisfactory means of accomplishing these operations is to place the crucible containing the wet filter paper in a cold electric muffle furnace and to turn on the current. Drying, charring, and ignition will usually occur at the desired rate.

(*b*) After ignition is complete, the crucible shall be allowed to cool to room temperature, and weighed. The use of a desiccator is not recommended.

(*c*) From the increase in weight of the crucible the percentage of sulfur shall be calculated from the formula:

$$\text{Percentage of sulfur} = \frac{\text{grams of } BaSO_4 \times 13.734}{\text{grams of oil used.}}$$

6. Duplicate determinations should agree to ± 2 per cent on the basis of the sulfur found. **Accuracy.**

TENTATIVE METHOD OF TEST

FOR

DETECTION OF FREE SULFUR AND CORROSIVE SULFUR COMPOUNDS IN GASOLINE.[1]

Serial Designation: D 130 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1922.

Scope.

1. This method of test shall be used for the detection of free sulfur and corrosive sulfur compounds in gasoline.

Procedure.

2. A clean strip of mechanically polished pure sheet copper about $\frac{1}{2}$ in. in width and 3 in. in length shall be placed in a suitable clean tube or sample bottle. Gasoline under test shall be added so that the copper strip is completely immersed. The test tube or sample bottle shall be closed with a loosely fitting cork and held in a suitable bath at 122° F. (50° C.).

At the end of three hours the gasoline exposed strip shall be removed and shall be compared with a similar strip of freshly polished copper.

Detection of Sulfur.

3. The presence of sulfur or corrosive sulfur compounds is indicated by the corrosion or discoloration of the gasoline exposed strip when compared with the fresh copper strip.

4. (*a*) Gasoline shall be reported as passing the test when on examination the exposed strip shows no discoloration as compared with the fresh copper strip.

(*b*) Gasoline shall be reported as not passing the test when on examination the exposed strip shows discoloration as compared with the fresh copper strip.

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

TENTATIVE METHOD OF TEST

FOR

PRECIPITATION NUMBER OF LUBRICATING OILS.[1]

Serial Designation: D 91 – 21 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921.

1. This method is commonly used for steam cylinder stocks and black oils and may be used for other lubricating oils. **Scope.**

DEFINITION.

2. *A.S.T.M. Precipitation Number of Lubricating Oils.*—The number of cubic centimeters of precipitate formed when 10 cc. of lubricating oil are mixed with 90 cc. of petroleum naphtha of definite quality and centrifuged under definite prescribed conditions. **Definition.**

APPARATUS.

3. *Centrifuge.*—The centrifuge shall be capable of whirling at least two 100-cc. centrifuge tubes filled with water at the required speed. It shall be of sound design and rugged construction so that it may be operated without danger. The tube carriers shall be so designed that the glass centrifuge tubes may be cushioned with water, rubber or other suitable material. The tube holders shall be surrounded during the operation by a suitable metal shield or case, strong enough to eliminate danger if any breakage occurs. **Centrifuge.**

Preferred forms of centrifuge shall have a diameter of swing (tip to tip of whirling tubes) of 15 to 17 in. and a speed of at least 1500 r.p.m. or equivalent. If the available centrifuge has a diameter of swing varying from these limits, it shall be run at the proper speed to give the same centrifugal force at the tips of the tubes as that obtained with the preferred form of centrifuge. The proper speed shall be calculated from the following formula in which D represents

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the diameter of swing (tip to tip of whirling tubes) of the centrifuge used:

$$\text{R.p.m.} = 1500\sqrt{\frac{16}{D}}$$

Centrifuge Tubes.

4. *Centrifuge tubes, A.S.T.M. type.*—These tubes shall be made of suitable glass and thoroughly annealed. The total capacity shall be about 125 cc. and the mouth shall be suitably constricted for closing with a cork. The graduations shall be clear and distinct, reading upward from the bottom of tube as follows:

RANGE.	SCALE DIVISIONS.	LIMIT OF ERROR.	NUMBERED.
0 – 3 cc.	0.1 cc.	0.05 cc.	1, 2, 3 cc.
3 – 5 "	0.5 "	0.2 "	4, 5 "
5 – 10 "	1.0 "	0.5 "	6, 8, 10 "
10 – 25 "	5.0 "	1.0 "	15, 20, 25 "
50 – 100 "	50.0 "	1.0 "	50, 100 "

The shape is optional provided it does not conflict with the other requirements.

Diluent.

5. (*a*) Petroleum naphtha meeting the following requirements shall be used for the diluent.

Specific gravity at 60° F. 0.695–0.705
Initial Boiling Point, A.S.T.M. .... 113–131° F. (45–55° C.)
End Point, A.S.T.M. .... not higher than 248° F. (120° C.)

(*b*) The initial boiling point and the end point of the petroleum naphtha shall be determined in accordance with the Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86 – 25 T) of the American Society for Testing Materials.[1]

PROCEDURE.

Preparing for Centrifuging.

6. Exactly 10.0 cc. of the oil to be tested shall be measured in each of two clean and dry centrifuge tubes at room temperature. Each tube shall be filled to the 100-cc. mark with the prescribed diluent and closed tightly with a softened cork (not a rubber stopper). Each tube shall then be inverted at least 20 times, allowing the liquid to drain thoroughly from the tapered tip of the tube each time. The tubes shall then be placed in a water bath at 90 to 95° F. for five minutes. The corks shall be momentarily removed to relieve any pressure and each tube shall again be inverted at least 20 times exactly as before. The success of this method depends to a large degree upon having a thoroughly homogeneous mixture which will

[1] See p. 325.

drain quickly and completely from the tapered tip when the tube is inverted.

7. The two centrifuge tubes shall then be placed in the centrifuge on opposite sides and shall be whirled at a rate of 1400 to 1500 r.p.m. or equivalent for 10 minutes. The volume of sediment at the bottom of each tube shall be read and recorded, estimating to 0.05 cc. if possible. The tubes shall then be replaced in the centrifuge, again whirled for 10 minutes as before, and removed for reading the volume of the sediment as before. This operation shall be repeated until the volume of sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings are required. **Centrifuging.**

8. The volume of the solid sediment at the bottom of each centrifuge tube shall be read, estimating to 0.1 cc. or closer if possible. If the two readings differ by not more than 0.1 cc. the mean of the two shall be reported as the "A.S.T.M. Precipitation Number." If the two readings differ by more than 0.1 cc. two more determinations shall be made and the average of the four determinations shall be reported. **Precipitation Number.**

ACCURACY.

9. With care and proper attention to details, duplicate determinations of Precipitation Number by this method should not differ by more than 0.1, provided the centrifuge tubes are accurate and readable to this degree. **Accuracy.**

TENTATIVE METHOD OF TEST

FOR

STEAM EMULSION OF LUBRICATING OILS[1]

Serial Designation: D 157 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

Issued, 1923

Scope. 1. This method shall be used on all oils when an emulsion, demulsibility or emulsification test is required. This method is commonly used for turbine oils and may be used for other lubricating oils.

Description. 2. The Modified Resistance to Emulsification Number (A.S.T.M. R.E.) is the number of minutes required for an oil to separate when emulsified and separated under definitely prescribed conditions.

I. APPARATUS

Steam Generator. 3. The steam generator shall be made of either metal or glass of at least one-liter capacity, capable of withstanding the heat necessary for continued use in the production of steam. It shall be fitted with three outlets with suitable connections for rubber tubing. In case of a metal generator, a large opening for filling and a suitable water gage shall be a necessary part of the apparatus.

Baths. 4. The baths shall be either of metal or glass, with a capacity of 3 to $3\frac{1}{2}$ liters and a depth of $7\frac{1}{2}$ to 9 in. A good quality battery jar or beaker is entirely satisfactory. A glass bath shall be used for emulsification and a metal or glass bath may be used for the separation.

Source of Heat. 5. Heat for the steam generator shall be supplied by a suitable gas burner or electric hot plate. The separating bath may be heated by any convenient means, including an auxiliary steam line as shown in Fig. 1.

Oil Container. 6. The oil container shall be a 25 by 200-mm. test tube graduated from zero or from 10 to 50 cc. in cubic centimeters, each even 5-cc. line to encircle the tube.

Steam Piping. 7. The steam piping or the steam delivery tube shall consist of a piece of thin-wall glass tubing, not less than 2.3 nor more than 2.7 mm. in inside diameter and 12 in. in length. The steam pipe shall be cut off diagonally at an angle of 30 deg. with the axis of the tube at

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

the discharge orifice and shall be bent at right angles 10 in. from the discharge orifice.

8. Accessories shall consist of: **Accessories.**

(*a*) Suitable wooden or metal frames or holders for holding all containers in a vertical position in the baths.

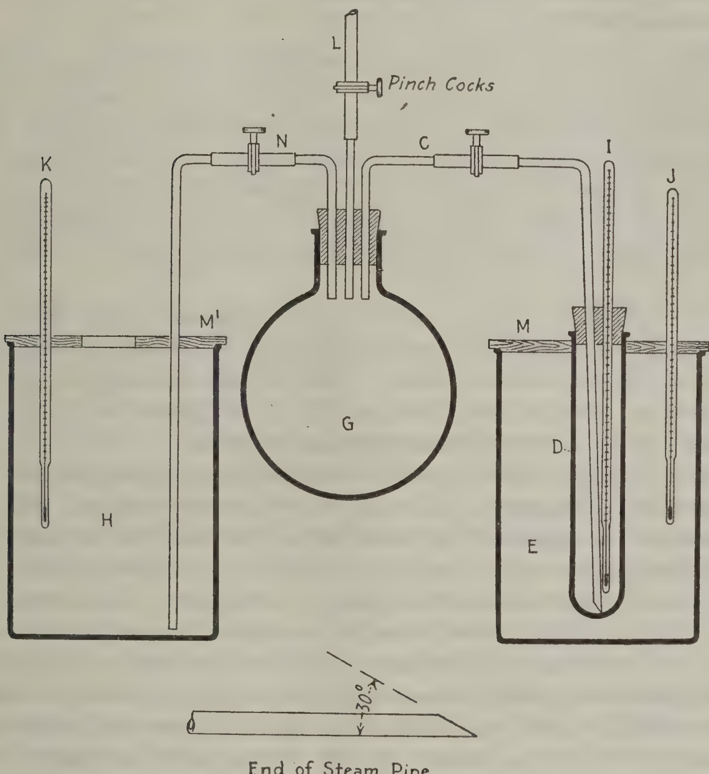

FIG. 1.—Apparatus for Test for Resistance to Emulsion. Self-Generating Steam Supply

(*b*) Thermometers for the separating and emulsifying baths (floating type thermometers of suitable range);

(*c*) Thermometers for the oil container tube (engraved-stem type, of suitable range, graduated in 1° F., 5 to 7 mm. in diameter);

(*d*) Corks, rubber tubing and screw pinch cocks.

II. PROCEDURE

9. The apparatus shall be assembled as shown in Fig. 1. The steam generator shall be filled one-half full of water and heat applied. **Preparation.**

The baths shall be filled with 3 liters ±60 cc. of water. The temperature in the separating bath shall be raised to and maintained at 200 to 203° F.

NOTE.—Care must be taken if glass battery jars are used, as direct heating by flame or electric hot point may cause breakage. Use of steam in this connection insures against breakage.

The temperature of the emulsifying bath shall be brought to not less than 67° F., nor more than 78° F. at the start of the test, and is not controlled thereafter. Twenty cubic centimeters of the oil to be rested shall be measured in the oil container at room temperature and the latter placed in the holder of the emulsifying bath. The steam pipe, or delivery tube, shall be connected to the steam generator with suitable rubber tubing, and screw pinch-cocks placed as show in Fig. 1.

Care shall be taken to see that the apparatus, particularly the oil container, oil container thermometer and the steam delivery tube are *chemically clean* before using. Care shall also be taken to prevent any foreign materials from entering the steam generator as any contamination of the steam renders the test valueless.

Emulsification.

10. The steam delivery tube line shall be steamed out until all condensation disappears. A cork having two openings, with the thermometer in one, shall be placed in the mouth of the oil container. The thermometer shall be adjusted so that the bottom of the bulb is $\frac{3}{4}$ to 1 in. from the bottom of the oil container. The steam delivery tube shall be inserted through the second opening in the cork (Note.—This fitting shall be loose.) so that the end of the steam delivery tube shall touch the center of the bottom of the oil container. Steam shall be admitted at a rate that will maintain the temperature of the oil, as shown on the thermometer in the oil container, between 190 and 195° F.

NOTE.—The usual time necessary for the temperature of the oil to come to this point is 45 to 75 seconds, depending on its character.

This control shall be effected by manipulation of the pinch-cocks on the steam delivery line and steam exhaust line from the steam generator. The steam supply shall be sufficient at all times to cause a generous discharge from the exhaust-line. Steaming shall be continued until the volume of condensed steam and oil in the oil container tube is 40 cc. ±3 cc. The time required for this operation shall be 4 to 6.5 minutes, depending on the quality of the oil, altitude, etc. If condensed water amounts to 20 cc. in less than 4 minutes, it shall

be taken as an indication of wet steam or incomplete steaming out of the line and the test shall be re-run.

NOTE.—The apparent volume in the tube near the end of the steaming operation is approximately 12 to 15 cc. greater than the actual volume due to displacement caused by steam, thermometer and steam delivery tube.

Separation. 11. The steam delivery tube shall be withdrawn as soon as the required volume is obtained. The oil container shall be transferred immediately to the separating bath which shall be maintained at 200 to 203° F. It is extremely important that the temperature of the separating bath be maintained within the given limits. A stop watch shall be started at the instant of withdrawal of the steam delivery tube. The cork containing the thermometer shall be removed after the oil container and contents have been placed in the separating bath. The contents of the oil container shall be examined every 30 seconds, and the volume of the separated oil layer shall be recorded. This examination shall be made by withdrawing the oil container and its contents from the separating bath, and reading the volumes.

NOTE.—This operation shall not require over five seconds.

No differentiation shall be made between clear and turbid oil.

This reading operation shall be continued until 20 cc. of oil have separated. In cases where the 20-cc. oil layer does not separate in 20 minutes, the test shall be discontinued at this point.

In cases where the interface between more-or-less clear oil and the emulsion is not a clear, straight horizontal line, the position of such a line is carefully estimated to the nearest 0.5 cc.

If, at the end of 20 minutes, there is not a well-defined horizontal interface, then, and then only, the contents of the oil container may be stirred for two seconds with a clean glass rod to obtain a better line of demarcation.

Interpretation. 12. In interpreting results, the assumption shall be made that the rate of emulsion separation is directly proportional to the Resistance to Emulsification of the tested oil. In order to establish a basis for comparison, a definite value of one shall be assigned to an oil which, under conditions of the test, separates completely from emulsion in one minute. This is equivalent to an emulsion separation rate of 0.33 cc. per second or 20 cc. per minute.

NOTE.—The expansion of the oil at the higher temperature is disregarded, so when the separation of the oil is complete, as regards the interface between the oil and emulsion layers, the volume of separated oil will always be more than 20 cc. This in no way interferes with the reproducibility of results, and the time shall be recorded when 20 cc.—and not necessarily all—of the oil have separated, when this occurs in less than 20 minutes.

A.S.T.M. R.E. Number.

13. Results shall be reported, in minutes and half minutes, of the time necessary for complete (20 cc.) oil separation. The time in minutes and half minutes shall be reported as the R.E. Number, A.S.T.M. Method. In cases where the required volume shall not have separated in 20 minutes, the oil shall be reported as having an R.E. Number, A.S.T.M. Method, of 20 plus. If duplicate determinations vary by more than one minute, a third test shall be made and the average of the three tests reported.

Note.—Oils which emulsify separate into three layers without exception: top, clear or turbid oil; middle, lacy or creamy emulsion; bottom, clear or milky water. The A.S.T.M. R.E. Number is derived from the top layer.

Reproducibility.

14. With care and proper attention to details, duplicate determinations of the R.E. Number, A.S.T.M. Method, should not differ by more than 0.5.

TENTATIVE METHOD OF TEST
FOR
SAPONIFICATION NUMBER.[1]

Serial Designation: D 94 – 21 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921.

SOLUTIONS REQUIRED.

Alcoholic Solution for Saponification.—Dissolve 58 g. of potassium hydrate "purified by alcohol" in 500 cc. of 95-per-cent purified ethyl alcohol. Allow the solution to settle in a dark place. Draw off the clear solution or filter through an asbestos filter and make up to one liter with 95-per-cent alcohol. The solution so prepared shall stand at least 20 hours before it is standardized.

Alcohol.—Purify 95-per-cent ethyl alcohol with silver oxide in the following manner:

Dissolve 1.5 g. of silver nitrate c.p. in about 3 cc. of water and add to 1 liter of alcohol in a glass stoppered bottle, and mix thoroughly. Dissolve 3 g. of potassium hydrate (by alcohol) in 10 to 15 cc. of warm alcohol. After cooling, add slowly to the alcoholic silver nitrate solution, stirring slightly. Allow the precipitated silver oxide to settle, siphon off the clear solution and distill on a steam bath.

Standard Hydrochloric Acid Solution.—One-half normal solution.

Phenolphthalein Solution.—One gram phenolphthalein in 100 cc. alcohol and water.

APPARATUS.

The saponification shall be carried out in a wide-mouthed flat-bottom extraction flask, or Erlenmeyer Flask, of 250 to 300-cc. capacity, fitted to a reliable condenser properly connected with a good cork. The boiling shall preferably be carried on by means of an electric hot plate.

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

Blank Determination.

Determination shall be made in duplicate in the alcoholic potash solution in the following manner:

Measure accurately into the flask 25 cc. of alcoholic potash solution from a calibrated pipette. The tip and outside of the pipette shall be wiped off with a clean filter paper before the solution is delivered. Then rinse out with 25 cc. of neutral alcohol. If a standard burette is used, allow 60 seconds total time for drawing and draining. Connect the flask to a suitable condenser and boil for three hours. Before disconnecting the flask, wash out the condenser with a few cubic centimeters of neutral alcohol; if a Soxhlet is used as the condenser the tip shall be washed off into the flask. Titrate while hot with N/2 HCl using three drops phenolphthalein indicator. The total number of cubic centimeters of N/2 HCl required for the blank represents the strength of the alcoholic potash solution.

Procedure.

For straight fats or oils use 2 to 3 g. of the material, for oils containing over 30-per-cent of fatty oils use about 5 g. and for oils containing less than 30-per-cent of fatty oils use about 10 g.

Weigh the oil accurately, by difference, from a small beaker into the saponification flask. Add 25 cc. of alcoholic potash solution and 25 cc. of neutral alcohol in the same manner as for the blank, connect to the condenser and boil for three hours. Titrate while hot. Calculate the Saponification Number from the difference between the number of cubic centimeters of N/2 HCl required for the determination and the average of the two blanks, using the formula:

$$\text{Saponification Number} = \frac{\text{Difference, cubic centimeters} \times 28.05}{\text{Weight of oil, grams}}.$$

Petrolic ether may be used with compounded cylinder oils in the quantity of 50 cc. providing a Soxhlet extraction flask is used to periodically collect this petrolic ether. The Soxhlet flask should be so adjusted, through the addition of glass rodding or beads when necessary, so that it will just overflow with the full quantity of petrolic ether.

The percentage of fatty oil (or fat) in a compounded petroleum product can be calculated from the saponification number of such a product only when the saponification number of the fatty oil is known. If the saponification numbers of both fatty oil and compounded oil are known, the following formula should be used:

$$\text{Percentage of fatty oil} = \frac{100 \times \text{Saponification Number of compounded oil.}}{\text{Saponification Number of fatty oil}}$$

For this determination the following values of Saponification Number may be used:

Fatty Oil.	Saponification Number.
Lard oil	192–198
Tallow	193–198
Neatsfoot	193–204
Fish	140–193
Sperm	120–140
Castor	176–187
Rapeseed	170–179
Soya bean	189–197
Peanut	186–197
Cottonseed	191–197
Blown rapeseed	195–216
Blown cottonseed	210–225
Degras	110–210

TENTATIVE METHOD OF TEST

FOR

NEUTRALIZATION NUMBER OF PETROLEUM PRODUCTS AND LUBRICANTS[1]

Serial Designation: D 188 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. The methods described herein are designed to indicate in petroleum products and lubricants and compounded products the presence of organic constitutents having acid characteristics and the contamination by alkalies and mineral acids.

Definitions. 2. (*a*) The Neutralization Number is the weight in milligrams of potassium hydroxide required to neutralize one gram of oil.

NOTE.—The Neutralization Number expresses the total amount of the mineral acid, and of the organic constituents having acid characteristics.

(*b*) The Alkali Neutralization Number is defined as the weight of acid required to neutralize one gram of oil, expressed in equivalent milligrams of potassium hydroxide.

(*c*) The Mineral Acid Neutralization Number is the number of milligrams of potassium hydroxide required to neutralize the mineral acid content in one gram of oil.

SOLUTIONS REQUIRED

3. (*a*) *Aqueous Potassium Hydroxide* (1 cc. = 5 mg. KOH).—Dissolve 5.1 g. of potassium hydroxide, c.p., in one liter of freshly boiled and cooled distilled water. Add a very small amount of barium hydroxide, sufficient to precipitate any potassium carbonate present. Standardize this solution against Bureau of Standards certified benzoic acid, using phenolphthalein as an indicator according to the relation:

$$\frac{5 \text{ mg. KOH}}{x \text{ mg. benzoic acid}} = \frac{56.104 \text{ g. KOH}}{122.048 \text{ g. benzoic acid}}$$

$$1 \text{ cc. of KOH} = 10.88 \text{ mg. benzoic acid.}$$

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

This weight of benzoic acid is required for standardization.

Make necessary adjustments so that the value of potassium hydroxide equals 5 mg. KOH per 1 cc.

NOTE.—Fit the solution bottle with a guard tube of soda lime to prevent access of carbon dioxide. The solution should be standardized at necessary intervals.

The weight of benzoic acid should be dissolved in 50 cc. of 95-per-cent alcohol and titrated cold. For blank, use same amount of alcohol and correct the titration.

(*b*) *Sulfuric Acid Solution* (1 cc. = 5 mg. KOH).—Match a sulfuric acid solution so that 1 cc. of the acid will be required to neutralize 1 cc. of the KOH, using phenolphthalein as an indicator, titrating in boiling solution.

(*c*) *Neutralized 95-per-cent Alcohol.*—Add a few drops of phenolphthalein and neutralize carefully the alcohol to a very faint pink end point with some of the above prepared alkali solution.

(*d*) *Phenolphthalein Indicator.*—Dissolve 10 g. of the indicator in 1 liter of 95-per-cent alcohol, preferably ethyl. Use 1 cc. of this strength for titration.

(*e*) *Methyl Orange Indicator.*—Dissolve 1 g. of the indicator in 1 liter of distilled water. Use 0.1 cc. of this strength for titration.

METHODS

4. *Method A.—Neutralization Number of Petroleum Products.*

Weight of Oil: Approximately 20 g. weighed accurately.

Volume of Solvent: 100 cc. of a mixture of 1:1 neutralized alcohol and distilled water.

Procedure: Agitate oil and solvent thoroughly and heat to boiling. Add 1 cc. of phenolphthalein indicator and titrate rapidly, with vigorous agitation, to a sharp pink end point. The titration must be completed in a hot solution, reheating same if found necessary.

The color change is noted in the alcohol water layer.

Calculation:

$$\frac{\text{(Cubic centimeters of KOH)} \times 5}{\text{Weight of oil taken}} = \text{mg. KOH per 1 g. of oil.}$$

5. *Method B.—Neutralization Number of Compounded Petroleum Products.*

Weight of Oil: Approximately 10 g. weighed accurately.

Volume of Solvent: 50 cc. of neutralized alcohol.

Procedure: Agitate oil and solvent thoroughly and heat to boiling. Add 1 cc. of phenolphthalein indicator and titrate rapidly, with vigorous agitation, to a sharp pink end point. The titration must be completed in a hot solution, reheating same if found necessary.

The color change is noted in the alcohol layer.

6. *Method C.—Alkali or Mineral Acid Neutralization Number in Non-compounded and compounded petroleum products.*

Weight of Oil: 25 to 50 g. weighed accurately.

Volume of Solvent: Boiling distilled water, 200 cc.

Procedure: Pour the oil and water in a 500-cc. separatory funnel. Shake vigorously and after oil and water have separated, drain water into a 500-cc. casserole. Add 100 cc. of boiling water to oil and agitate. Drain separated water to casserole. Repeat washing and to the accumulated 100 cc. of water add 1 drop of phenolphthalein. Boil. If solution turns pink add 1 cc. of the indicator and titrate with the sulfuric acid solution until extraction is colorless.

If the addition of 1 drop of phenolphthalein causes no change in color, add 0.1 cc. of methyl orange. If color changes to red or pink, titrate with the KOH solution until the red color is just discharged.

An equal volume of distilled water shall be titrated with the standard solution used, and the amount of alkali or acid required shall be subtracted from the titration for blank correction.

Calculation: If the acid solution was required to discharge the phenolphthalein end point, the alkali neutralization number equals:

$$\frac{(\text{Cubic centimeters of acid}) \times 5}{\text{Weight of oil}}$$

If the alkali solution was required to discharge the methyl orange end point, the mineral acid neutralization number equals:

$$\frac{(\text{Cubic centimeters of alkali}) \times 5}{\text{Weight of oil}}$$

TENTATIVE METHOD OF TEST

FOR

CARBON RESIDUE OF LUBRICANTS[1]

Serial Designation: D 189 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924.

CARBON RESIDUE[2]

(Conradson Method)

Apparatus.

1. The apparatus (see Fig. 1) consists of:

(*a*) Porcelain crucible, wide form, glazed throughout, 25 to 26-cc. capacity, 46 mm. in diameter.

(*b*) Skidmore iron crucible, 45-cc. ($1\frac{1}{2}$-oz.) capacity, 65 mm. in diameter, 37 to 39 mm. high with cover, without delivery tubes and one opening closed.

(*c*) Wrought-iron crucible with cover, about 180 cc. capacity, 80 mm. diameter, 58 to 60 mm. high. At the bottom of this crucible a layer of sand is placed about 10 mm. deep or enough to bring the Skidmore crucible with cover on nearly to the top of the wrought-iron crucible.

(*d*) Triangle, pipe stem covered, projection on side so as to allow flame to reach the crucible on all sides.

(*e*) Sheet iron or asbestos hood provided with a chimney about 2 to $2\frac{1}{2}$ in. high, $2\frac{1}{8}$ to $2\frac{1}{4}$ in. in diameter to distribute the heat uniformly during the process.

(*f*) Asbestos or hollow sheet iron block, 6 to 7 in. square, $1\frac{1}{4}$ to $1\frac{1}{2}$ in. high, provided with opening in center $3\frac{1}{4}$ in. in diameter at the bottom, and $3\frac{1}{2}$ in. in diameter at the top.

Procedure.

2. The test shall be conducted as follows:

Ten grams of the oil to be tested are weighed in the porce-

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

[2] This method is a modification by P. H. Conradson of his original method and apparatus for Carbon Test and Ash Residue in Petroleum Lubricating Oils. See *Proceedings*, Eighth International Congress of Applied Chemistry, New York, September, 1912, Vol. 1, p. 131; also reprint in the *Journal of Industrial and Engineering Chemistry*, Vol. 4, No. 11, November, 1912.

lain crucible, which is placed in the Skidmore crucible and these two crucibles set in the larger iron crucible, being careful to have the Skidmore crucible set in the center of the iron crucible, covers being applied to the Skidmore and iron crucibles.

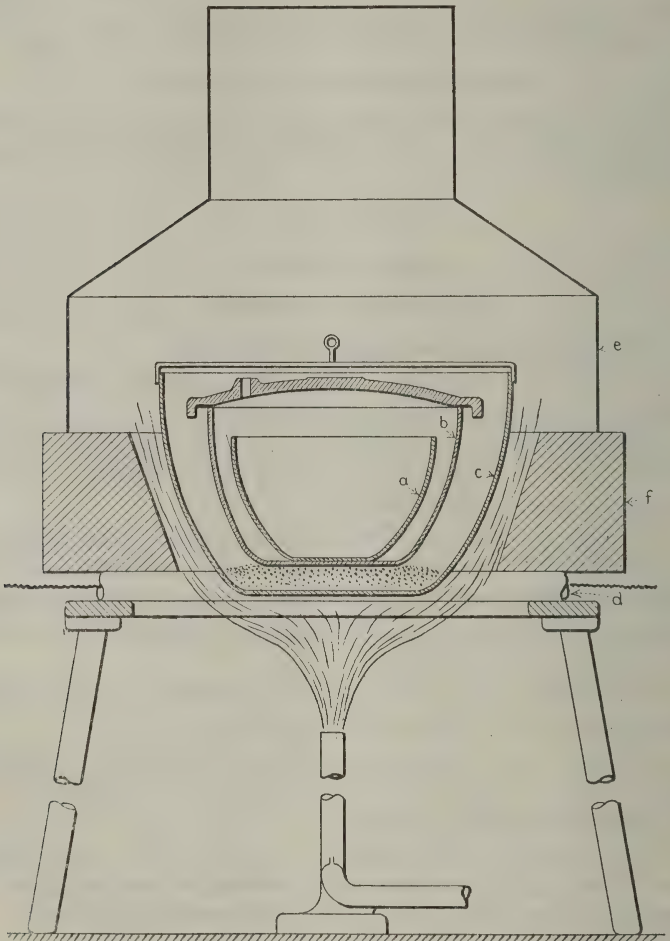

FIG. 1.—Apparatus for Determining Carbon Residue.

Place on triangle and suitable stand with asbestos block and cover with sheet iron or asbestos hood in order to distribute the heat uniformly during the process.

Heat from a Bunsen burner or other burner is applied with a high flame surrounding the large crucible, as shown in

Fig. 1, until vapors from the oil start to ignite over the crucible, when the heat is slowed down so that the vapor (flame) will come off at a uniform rate. The flame from the ignited vapors should not extend over 2 in. above the sheet iron hood. After the vapor ceases to come off the heat is increased as at the start and kept so for five minutes, making the lower part of large crucible red hot, after which the apparatus is allowed to cool somewhat before uncovering the crucible. The porcelain crucible is removed, cooled in a desiccator and weighed.

The entire process should require about one-half hour to complete when heat is properly regulated. The time will depend somewhat upon the kind of oil tested, as a very thin, rather low-flash-point oil will not take as long as a heavy, thick, high-flash-point oil.

TENTATIVE METHOD OF TEST

FOR

CLOUD AND POUR POINTS OF PETROLEUM PRODUCTS[1]

Serial Designation: D 97 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1922, 1923, 1925.

Cloud Point. 1. (*a*) The Cloud Point of a petroleum oil is that temperature at which paraffin wax or other solid substances begin to crystallize out or separate from solution when the oil is chilled under certain definite specified conditions.

Pour Point. (*b*) The Pour Point of a petroleum oil is the lowest temperature at which this oil will pour or flow when it is chilled without disturbance under certain definite specified conditions.

Scope. 2. (*a*) The test for cloud point shall be used only for oils which are transparent in layers $1\frac{1}{2}$ in. thick.

(*b*) The test for pour point shall be used for all other petroleum oils and may be used for oils on which the test for cloud point is permitted.

I. APPARATUS

(See Fig. 1)

Test Jar. 3. The test jar, *a*, shall be of clear glass, cylindrical form, flat bottom, approximately $1\frac{1}{4}$ in. in inside diameter and $4\frac{1}{2}$ to 5 in. high. An ordinary 4-oz. oil sample bottle may be used if the test jar is not available.

Thermometers. 4. The thermometer, *b*, shall conform to the requirements of one of the following specifications:

(*a*) These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being -38 to $+50°$ C. or -36 to $+120°$ F., respectively.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: -38 to $+50°$ C. in 1° C. or -36 to $+120°$ F. in 2° F.

TOTAL LENGTH: 220 to 224 mm. (8.69 to 8.81 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 7.0 to 8.0 mm. (0.28 to 0.31 in.).

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

BULB: Corning normal or equally suitable thermometric glass.
Length, not over 9.5 mm. (0.37 in.).
Diameter, not greater than stem.

DISTANCE TO −38° C. OR −36° F. LINE FROM BOTTOM OF BULB: 120 to 130 mm. (4.73 to 5.12 in.).

DISTANCE TO +49° C. OR +120° F. LINE FROM TOP OF THERMOMETER: 19 to 25 mm. (0.75 to 0.98 in.).

EXPANSION CHAMBER: To permit heating to 100° C. or 212° F.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Plain.

GRADUATION: All lines, figures, and letters clear cut and distinct. Lines at multiples of 5° C. or 10° F. to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

IMMERSION: 108 mm. or $4\frac{1}{4}$ in. The words "108-mm. immersion" on Centigrade thermometers or "$4\frac{1}{4}$-in. immersion" on Fahrenheit thermometers and a line around the stem 108.0 mm. or 4.25 in. above the bottom of the bulb shall be etched on the thermometer.

SPECIAL MARKING: "A.S.T.M. Cloud and Pour," a serial number and the manufacturer's name or trade mark shall be etched on the stem.

SCALE ERROR: The error at any point of the scale, when the thermometer is standardized as provided below, shall not exceed 0.5° C. or 1° F.

STANDARDIZATION: The thermometer shall be standardized at the ice point and at intervals of approximately 20° C. or 40° F. for 108-mm. or $4\frac{1}{4}$-in. immersion and for an average temperature of 21° C. or 70° F. for the emergent mercury column.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M.Cloud and Pour, −38 to +50° C." or "A.S.T.M. Cloud and Pour, −36 to +120° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

(*b*) These specifications cover a special low cloud and pour test thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being −60 to +20° C. or −70 to +70° F.

TYPE: Etched stem, glass.

LIQUID: Toluene or other suitable liquid colored red with a permanent dye.

RANGE AND SUB-DIVISION: −60 to +20° C. in 1° C. or −70 to +70° F. in 2° F.

TOTAL LENGTH: 227 to 231 mm. (8.92 to 9.08 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter 7.0 to 8.0 mm. (0.28 to 0.31 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 8.0 to 9.5 mm. (0.31 to 0.37 in.).
Diameter, 5.0 to 6.5 mm. (0.22 to 0.26 in.).

DISTANCE TO −57° C. or −70° F. LINE FROM BOTTOM OF BULB: 120 to 130 mm. (4.73 to 5.12 in.).

Distance to +20° C. or +68° F. Line from Top of Thermometer: 35 to 45 mm. (1.38 to 1.77 in.).

Expansion Chamber: To permit heating to +60° C. or +140° F.

Filling above Liquid: Gas under pressure.

Top Finish: Plain.

Graduation: All lines, figures and letters clear cut and distinct, lines at multiples of 5° C. or 10° F. to be longer than the remaining lines. Graduations to be numbered at each multiple of 10° C. or 20° F.

Immersion: 108 mm. ($4\frac{1}{4}$ in.). The words "108-mm. immersion" on Centigrade thermometers, or "$4\frac{1}{4}$-in. immersion" on Fahrenheit thermometers and a line around the stem 108 mm. (4.25 in.) above the bottom of the bulb, shall be etched on the thermometer.

Special Marking: "A.S.T.M. Low Cloud and Pour" a serial number and the manufacturer's name or trade mark shall be etched on the thermometer.

Scale Error: The error at any point of the scale when the thermometer is standardized as provided below, shall not exceed 1° C. or 2° F.

Standardization: The thermometer shall be standardized at the ice point and at intervals of approximately 20° C. or 35° F. for 108-mm. or $4\frac{1}{4}$-in. immersion, and for an avergae temperature of 21° C. or 70° F. for the emergent liquid column.

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking "A.S.T.M. Low Cloud and Pour, −60 to +20° C." or A.S.T.M. Low Cloud and Pour, −70 to +70° F.," according to the type of thermometer.

Note.—For the purpose of interpreting these specifications, the following definitions apply:

The total length is the overall length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

Cork. 5. The cork, *c*, shall fit the test jar, and shall be bored centrally to take the test thermometer.

Jacket. 6. The jacket, *d*, shall be of glass or metal, shall be water tight, of cylindrical form, flat bottom, about $4\frac{1}{2}$ in. deep, with inside diameter $\frac{1}{2}$ in. greater than outside diameter of the test jar.

Cork Disk. 7. A disk of cork or felt, *e*, $\frac{1}{4}$ in. thick and of the same diameter as the inside of the jacket will be required.

Gasket. 8. The ring gasket, *f*, shall be about $\frac{3}{16}$ in. thick, and made to fit snugly around the outside of the test jar and loosely inside the jacket. This gasket may be made of cork, felt or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape. The purpose of the ring gasket is to prevent the test jar from touching the jacket.

Bath. 9. The cooling bath, *g*, shall be of a type suitable for obtaining the required temperatures. The size and shape of the bath are optional but a support, suitable for holding the jacket firmly in a

vertical position, is essential. For determination of very low pour points, a smaller insulated cooling bath may be used and the test jar placed directly in it. The required bath temperatures may be

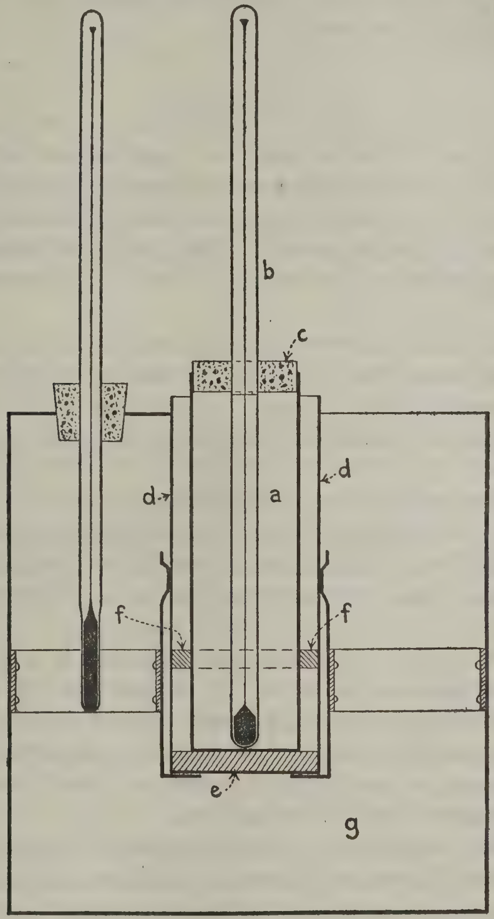

FIG. 1.—Apparatus for Cloud and Pour Test
(as assembled for Cloud Test)

maintained by refrigeration if available, otherwise by suitable freezing mixtures.

NOTE.—The freezing mixtures commonly used are as follows:

For temperatures down to	50° F.,	ice and water.
" " " "	10° F.,	crushed ice and sodium chloride.
" " " "	−15° F.,	crushed ice and calcium chloride.
" " " "	−70° F.,	solid carbon dioxide and acetone or gasoline.

The last named mixture may be made as follows: In a covered metal beaker chill a suitable amount of acetone to 10° F., or lower, by means of an ice-salt mixture. Invert a cylinder of liquid carbon dioxide and draw off carefully into a chamois skin bag the desired amount of carbon dioxide, which through rapid evaporation will quickly become solid. Then add to the chilled acetone enough of the solid carbon dioxide to give the desired temperature.

II. PROCEDURE

Procedure for Cloud Point. 10. The oil to be tested shall be brought to a temperature at least 25° F. above the approximate cloud point. Moisture, if present, shall be removed by any suitable method, as by filtration through dry filter paper until the oil is perfectly clear, but such filtration shall be made at a temperature at least 25° F. above the approximate cloud point.

The clear oil shall be poured into the test jar, *a*, to a height of not less than 2 nor more than 2¼ in. The test jar may be marked to indicate the proper level.

The test jar shall be tightly closed by the cork, *c*, carrying the test thermometer, *b*, in a vertical position in the center of the jar with the thermometer bulb resting on the bottom of the jar.

The disk, *e*, shall be placed in the bottom of the jacket, *d*, and the test jar with the ring gasket, *f*, 1 in. above the bottom shall be inserted into the jacket. The disk, jacket and inside of jacket shall be clean and dry.

The temperature of the cooling bath, *g*, shall be adjusted so that it is below the cloud point of the oil by not less than 15 nor more than 30° F. and this temperature shall be maintained throughout the test. The jacket, containing the test jar, shall be supported firmly in a vertical position in the cooling bath so that not more than 1 in. of the jacket projects out of the cooling medium.

At each test thermometer reading which is a multiple of 2° F., the test jar shall be removed from the jacket, quickly but without disturbing the oil, inspected for cloud, and replaced in the jacket. This complete operation shall require not more than three seconds.

When such inspection first reveals a distinct cloudiness or haze in the oil at the bottom of the test jar, the reading of the test thermometer, corrected for error if necessary, shall be recorded as the cloud point.

11. The oil shall be poured into the test jar, *a*, to a height of not less than 2 nor more than 2¼ in. When necessary, the oil shall be heated in a water bath just sufficiently for pouring into the test jar.

The test jar shall be tightly closed by the cork, *c*, carrying the test thermometer, *b*, in a vertical position in the center of the jar with the thermometer bulb immersed so that the beginning of the capillary shall be $\frac{1}{8}$ in. below the surface of the oil.

Heat without stirring to a temperature of 115° F. in a bath maintained at not higher than 118° F. The oil shall then be cooled to 90° F. in air or in a water bath approximately 77° F. in temperature. Oils with which the low cloud and pour test thermometer can be used from the beginning of the test shall be cooled to 60° F. in any convenient manner before placing that thermometer in position.

The disk, *e*, shall be placed in the bottom of the jacket, *d*, and the test jar, with the ring gasket, *f*, 1 in. above the bottom, shall be inserted into the jacket. The disk, gasket and inside of jacket shall be clean and dry.

After the oil has cooled enough to allow the formation of paraffin wax crystals, great care shall be taken not to disturb the mass of the oil nor to permit the thermometer to shift in the oil. Any disturbance of the spongy network of wax crystals will lead to low and fictitious results.

The temperature of the cooling bath, *g*, shall be adjusted so that it is below the pour point of the oil by not less than 15 nor more than 30° F. and this temperature shall be maintained throughout the test. The jacket, containing the test jar, shall be supported firmly in a vertical position in the cooling bath so that not more than 1 in. of the jacket projects out of the cooling medium.

Beginning at a temperature 20° F. before the expected pour point, at each test thermometer reading which is a multiple of 5° F., the test jar shall be removed from the jacket carefully and shall be tilted just sufficiently to ascertain whether there is a movement of the oil in the test jar. The complete operation of removal and replacement shall require not more than three seconds. As soon as the oil in the test jar does not flow when the jar is tilted, the test jar shall be held in a horizontal position for exactly five seconds, as noted by a stop watch or other accurate timing device and observed carefully. If the oil shows any movement under these conditions, the test jar shall be immediately replaced in the jacket and the same procedure repeated at the next temperature reading 5° F. lower.

The test shall be continued in this manner until a point is reached at which the oil in the test jar shows no movement when the test jar is held in a horizontal position for exactly five seconds. Certain lubricating oils tend to move as a whole and should be very closely observed. The reading of the test thermometer at this

temperature, corrected for error if necessary, shall be recorded. The pour point shall be taken as the temperature 5° F. above this solid point.

Special Procedure for Black Oils and Cylinder Stocks

12. (*a*) In those cases where it is known that a sample has been subjected to some temperature higher than 115° F. during the preceding 24 hours, or where the history of the sample in this respect is not known, the sample shall be held in the laboratory 24 hours before testing, unless threee consecutive tests of the same sample in the same test jar check results. For these particular oils this shall be called the maximum pour point.

(*b*) The minimum pour point shall be determined by heating a sample with stirring to 220° F. The oil shall then be poured in the test jar, cooled to 90° F. as before, and the pour point determined as described in Section 11.

(*c*) The pour point of such oils shall be reported as lying between the maximum and minimum pour points (*e. g.*, Pour Point 35°/50° F.).

TENTATIVE METHODS OF TESTING GAS OILS

(GRAVITY, DISTILLATION, SULFUR, CARBON RESIDUE, POUR POINT, VISCOSITY, WATER)[1]

Serial Designation: D 158 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1925.

GRAVITY

1. The gravity shall be determined at 60°/60° F. (15.6°/15.6° C.) in a pyknometer, accurately standardized, or by means of a hydrometer or Westphal balance, the accuracy of which has been verified. The gravity shall be recorded as specific gravity or gravity, degrees A.P.I., at 60°/60° F. (15.6°/15.6° C.). **Gravity.**

DISTILLATION

APPARATUS

2. *Flask.*—The distillation flask shall be a Saybolt distilling flask, the dimensions and permissible variations being as follows: **Flask.**

	Centimeters	Inches	Tolerances, cm.
Diameter of bulb, outside	8.57	3.37	0.3
Diameter of neck, inside	1.50	0.59	0.1
Over all length, neck plus bulb	21.50	8.46	0.3
Length of vapor tube	17.50	6.89	0.5
Diameter of vapor tube, outside	0.60	0.24	0.05
Diameter of vapor tube, inside	0.40	0.16	0.05
Thickness of vapor tube wall	0.10	0.04	0.02

The center of the vapor tube shall be 6.5 cm. (2.56 in.), ±0.3 cm., below the top of the neck. The tube is approximately in the middle of the neck and set at an angle of 75 deg., ±3 deg., with the vertical.

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 250 Park Ave., New York City.

Condenser. 3. *Condenser.*—The condenser[1] shall consist of a $\frac{9}{16}$-in. (14.29-mm.), outside diameter, No. 20 Stubbs Gage seamless brass tube, 22 in. (55.88 cm.) in length. It shall be set at an angle of 75 deg. with the vertical and shall be surrounded with a bath 15 in. (38.1 cm.) in length, approximately 4 in. (10.16 cm.) in width by 6 in. (15.24 cm.) in height. The bath shall be provided with suitable connections for circulating water through it. The lower end of the condenser tube shall be cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the receiving graduate at a point approximately 1 to $1\frac{1}{4}$ in. (2.54 to 3.18 cm.) below the top of the graduate when it is in position to receive the distillate.

Shield. 4. *Shield.*—The shield[2] shall be made of approximately No. 22 gage sheet metal and shall be 19 in. (48.26 cm.) in height, 11 in. (27.94 cm.) in length and 8 in. (20.32 cm.) in width, with a door on one narrow side, with two openings, 1 in. (2.54 cm.) in diameter, equally spaced, in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings shall be $8\frac{1}{2}$ in. (21.59 cm.) below the top of the shield. There shall also be three $\frac{1}{2}$-in. (1.27-cm.) holes in each of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield.

Support. 5. *Ring Support and Hard Asbestos Boards.*—The ring support may be of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and shall be supported on a stand inside the shield. There shall be two hard asbestos boards: One 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.) with a hole $2\frac{3}{4}$ in. (6.99 cm.) in diameter in its center, the sides of which shall be perpendicular to the surface; the other, an asbestos board to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These shall be arranged as follows: The second asbestos board shall be placed on the ring and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat shall be applied to the flask only through the $2\frac{3}{4}$-in. (6.99-cm.) opening in the first asbestos board.

Heater. 6. *Gas Burner.*—The burner shall be so constructed that sufficient heat can be obtained to distill the product at the uniform rate

[1] A condenser conforming to these specifications is illustrated in the Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86 – 25 T) of the American Society for Testing Materials. See p. 325.

[2] A shield conforming to these specifications is illustrated in the Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86 – 25 T) of the American Society for Testing Materials. See p. 325.

specified below. The flame should never be so large that it spreads over a circle of diameter greater than $4\frac{1}{2}$ in. (11.43 cm.) on the under surface of the asbestos board. A sensitive regulating valve and gas pressure governor are desirable adjuncts, as they give complete control of heating.

7. *Thermometer.*—The A.S.T.M. High Distillation Thermometer, conforming to the requirements specified in Section 6 (*b*) of the Tentative Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (Serial Designation: D 86 – 25 T) of the American Society for Testing Materials,[1] shall be used. **Thermometer.**

8. *Graduates.*— **Graduates.**

(*a*) *For Sample.*—A graduate or pipette graduated to deliver 200 cc., ± 1.0 cc., may be used for measuring the sample to be tested.

(*b*) *For Fractions.*—The graduates used for measuring fractions shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 cc. and the graduated portion shall be not less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) in length. It shall be graduated in single cubic centimeters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom at intervals of 10 cc. The overall height of the graduate shall be not less than $9\frac{3}{4}$ in. (24.8 cm.) nor more than $10\frac{1}{4}$ in. (26.0 cm.). The graduations shall not be in error by more than 1 cc. at any point on the scale.

PROCEDURE

9. (*a*) Provision shall be made for circulating water through the condenser. **Procedure.**

(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft, lint-free cloth attached to a cord or copper wire may be used for this purpose.

(*c*) A volume of water-free oil equivalent to 200 cc. at 55 to 65° F. (12.8 to 18.3° C.) shall be placed in the flask.

(*d*) The thermometer, provided with a cork, shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(*e*) The charged flask shall be placed over the $2\frac{3}{4}$-in. (6.99-cm.) opening in the asbestos board with the vapor outlet tube inserted

[1] See p. 325.

into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.).

(*f*) A clean, dry graduated cylinder shall be placed at the outlet of the condenser tube in such position that the condenser tube shall extend into the graduate approximately 1 in. (2.54 cm.) but not below the 100-cc. mark.

Distillation.

10. When everything is in readiness, water shall be circulated through the condenser bath at such a rate that the water overflowing the condenser shall be at a temperature of 90 to 100° F. (32.2 to 37.8° C.). Heat shall be applied to the contents of the flask at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 10 nor more than 15 minutes. When the first drop falls from the end of the condenser, the reading of the distillation thermometer shall be recorded as the *initial boiling point.* The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation will proceed at a uniform rate of not less than 8 cc. nor more than 10 cc. per minute. A fraction shall be separated at every temperature point above the initial boiling point that is a multiple of 50° F.[1] When the temperature of the distilling vapors reaches 500° F. (260° C.), the temperature of the water in the condenser bath shall be adjusted to approximately 140° F. and the bath maintained between 140 and 160° F. (60 to 71.1° C.) for the remainder of the test. The distillation shall be continued until a vapor temperature of 700° F. (371.1° C.) is reached or until the maximum temperature point is reached. The maximum temperature point may result from complete distillation of the oil below 700° F. (371.1° C.), or from cracking.[2]

In case the oil distills completely below 700° F. (371.1° C.), the

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[1] When it is agreed to make allowance for the effect of barometric pressure, the observed temperature points at which fractions are to be separated in order to conform to multiples of 50° F. at 760 mm. shall be calculated by the Sydney Young equation, so expressed as to simplify the application:

$$C_f = 0.00012\ P(460 + t_f),$$

in which C_f is the correction to be added to the observed temperature when the barometer is above 760 mm. and to be subtracted from the observed temperature when the barometer is below 760 mm.

P is the difference in millimeters of mercury (at 32° F.) between 760 mm. and the observed barometric pressure, corrected for instrumental errors.

t_f is the temperature point to be corrected.

[2] Cracking will be evidenced by an increase in distilling rate with a thermometer reading that may advance very slowly, remain stationary, or recede, and an effort to adjust the distilling rate will usually result in a decided drop in the temperature reading. When this condition is observed, it is advisable to discontinue the distillation.

final adjustment of heat shall be made when a quantity of distillate amounting to 90 per cent of the sample has been collected. The heating shall be continued until the mercury reaches a maximum height and starts to fall consistently; but in no case should the period between the 90-per-cent point and the maximum temperature be more than 10 minutes. The highest temperature observed on the distillation thermometer shall be recorded as the *maximum temperature.*

Sufficient time shall be allowed after distillation is discontinued for the condenser to drain into the final fraction. Each fraction shall be brought to room temperature, the volume read and recorded, and the fraction set aside for the determination of the gravity. The percentage of distillate in each fraction shall be calculated and recorded. The difference between 100 and the sum of the distillates in per cent shall be calculated and recorded as *residue and loss.*

The gravity of each fraction and of the residue shall be determined at or corrected to 60° F. (15.6° C.). The gravity of each fraction shall be recorded as specific gravity or as gravity, degrees A. P. I.

SULFUR

11. The sulfur shall be determined in accordance with the Tentative Method of Test for Sulfur in Petroleum Oils Heavier than Illuminating Oil (Serial Designation: D 129 – 22 T) of the American Society for Testing Materials.[1] **Sulfur.**

CARBON RESIDUE

12. The carbon residue shall be determined in accordance with the Conradson Method, as described in the Tentative Method of Test for Carbon Residue of Lubricants (Serial Designation: D 189 – 24 T) of the American Society for Testing Materials.[2] **Carbon Residue.**

POUR POINT

13. The pour point shall be determined in accordance with the Tentative Method of Test for Cloud and Pour Points of Petroleum Products (Serial Designation: D 97 – 25 T) of the American Society for Testing Materials.[3] **Pour Point.**

[1] See p. 391.
[2] See p. 409.
[3] See p. 412.

VISCOSITY

Viscosity. 14. The viscosity shall be determined on the Saybolt Universal Viscosimeter at 100° F. in accordance with the Tentative Methods of Test for Viscosity of Petroleum Products and Lubricants (Serial Designation: D 88 – 25 T) of the American Society for Testing Materials.[1]

WATER

Water. 15. Water shall be determined in accordance with the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[2]

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[1] See p. 358.

[2] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

COMMERCIAL SIZES OF BROKEN STONE AND BROKEN SLAG FOR HIGHWAY CONSTRUCTION.[1]

Serial Designation: D 63 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1920; REVISED, 1923.

Material Covered.

1. These specifications cover the standard size designations and maximum permissible range in mechanical analyses for nine commercial grades of broken stone and broken slag, when used in the construction of plain or bituminous macadam, bituminous concrete, sheet asphalt and cement-concrete roads and pavements.

I. MANUFACTURE.

Screening Process.

2. The standard sizes herein specified may be produced from the run of the crusher by means of any screening process in which the factors influencing the efficiency of the screening operation are so adjusted that the designated sizes will conform to the requirements for mechanical analysis specified in Section 4.

II. STANDARD SIZES.

Standard Sizes.

3. Each of the standard sizes shall be designated by reference to both the upper and lower nominal limiting size of the product: thus, "$\frac{1}{4}$ to $\frac{3}{4}$-in. size" indicating that portion of the product of a crusher, all of which is nominally between $\frac{1}{4}$ and $\frac{3}{4}$ in. in size.

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

III. PERMISSIBLE VARIATIONS IN MECHANICAL ANALYSIS.

Permissible Variations in Mechanical Analysis.

4. The designated size for each grade together with the corresponding maximum permissible variations in mechanical analyses as determined by the use of laboratory screens, are given in the following table:

Maximum Permissible Range in Mechanical Analysis for Each Size
Percentage by Weight Passing Laboratory Screens.

Designated Size, in.	Diameter of Screen Openings, in.						
	$\frac{1}{4}$	$\frac{1}{2}$	$\frac{3}{4}$	1	$1\frac{1}{4}$	$2\frac{1}{2}$	$3\frac{1}{2}$
$0 - \frac{1}{4}$..........	85–100	......	......	......	......	......	......
$0 - \frac{1}{2}$..........	0– 75	95–100	......	......	......	......	......
$0 - \frac{3}{4}$..........	40– 80	......	95–100	......	......	......	......
$\frac{1}{4} - \frac{3}{4}$..........	0– 15	25– 75	95–100	......	......	......	......
$\frac{1}{4} - 1\frac{1}{4}$..........	0– 5	......	25– 75	......	95–100	......	......
$\frac{1}{4} - 2\frac{1}{2}$..........	0– 5	......	......	......	25– 75	95–100	......
$\frac{3}{4} - 1\frac{1}{4}$..........	......	......	0– 15	25– 75	95–100	......	......
$1\frac{1}{4} - 2\frac{1}{2}$..........	......	......	......	......	0– 15	95–100	......
$2\frac{1}{2} - 3\frac{1}{2}$[a]..........	......	......	......	......	......	0– 15	95–100

[a] In the case of light or porous slags a 4-in. maximum size may be specified instead of $3\frac{1}{2}$ in.

IV. SIZE OF SAMPLES.

Samples.

5. Samples of broken stone or broken slag which are to be examined to determine conformity with these specifications shall weigh at least 50 times the weight of the largest piece therein and be strictly representative of the product sampled.

Explanatory Statement.

The commercial sizes designated in the accompanying proposed specifications provide primarily for the separation of the run of crusher into five sizes not including the 0 to $\frac{1}{2}$-in. size. Although the limiting sizes were determined after carefully considering the size requirements of the various types of roads using broken stone or slag, no definite requirements regarding use have been inserted, as such requirements will be included in specifications which are to be prepared by Committee D-4. It will be noted that a general tolerance of 15 per cent on the lower limiting size and 5 per cent on the upper limiting size for each grade is specified. It is felt that this will cover all unavoid-

able variations in grading of a given designated product. An intermediate size requirement of from 25 to 75 per cent is also given for the $\frac{1}{4}$ to $\frac{3}{4}$-in. and the $\frac{3}{4}$ to $1\frac{1}{4}$-in. sizes. It is felt that for the two larger sizes an intermediate size requirement is not necessary. For the combination sizes which will ordinarily be used in cement-concrete work, a lower tolerance of 5 per cent is specified. In Table I various uses to which the different grades are adapted are indicated.

TABLE I.—VARIOUS USES FOR WHICH THE DIFFERENT GRADES OF STONE ARE ADAPTED.

Designated Size, in.	Suggested Uses.
$0 - \frac{1}{4}$.....	For fine screenings for waterbound macadam.
$0 - \frac{1}{2}$.....	" fine aggregate for bituminous concrete.
$\frac{1}{4} - \frac{3}{4}$.....	" dustless screenings or chips for bituminous macadam or bituminous surface treatment.
$\frac{3}{4} - 1\frac{1}{4}$.....	" coarse chips or 1-in. stone for bituminous macadam.
$1\frac{1}{4} - 2\frac{1}{2}$.....	" wearing course, waterbound macadam or bituminous macadam.
$2\frac{1}{2} - 3\frac{1}{2}$.....	" broken-stone base course.
	COMBINATION SIZES.
$0 - \frac{3}{4}$.....	For coarse screenings for waterbound macadam.
$\frac{1}{4} - 1\frac{1}{4}$.....	" aggregate for reinforced concrete, bituminous concrete, sheet asphalt binder course.
$\frac{1}{4} - 2\frac{1}{2}$.....	" coarse aggregate for cement-concrete roads and structures.

TENTATIVE SPECIFICATIONS

FOR

COMMERCIAL SIZES OF SAND AND GRAVEL FOR HIGHWAY CONSTRUCTION.[1]

Serial Designation: D 64-20 T.

This is a **Tentative Standard** only, published for the purpose of eliciting critcism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1920.

Material Covered.

1. These specifications cover the standard size designations and maximum permissible range in mechanical analyses for nine commercial grades of sand and gravel, when used in the construction of waterbound-gravel and cement-concrete roads and pavements.

I. MANUFACTURE.

Screening Process.

2. The standard sizes herein specified may be produced from any screening process in which the factors influencing the efficiency of the screening operation are so adjusted that the designated sizes will conform to the requirements for mechanical analysis specified in Section 4.

II. STANDARD SIZES.

Standard Sizes.

3. Each of the standard sizes shall be designated by reference to both the upper and lower nominal limiting size of the product: thus, "$\frac{1}{4}$ to $\frac{3}{4}$-in. size" indicating that portion of the product of a sand and gravel screening plant, all of which is nominally between $\frac{1}{4}$ and $\frac{3}{4}$ in. in size.

III. PERMISSIBLE VARIATIONS IN MECHANICAL ANALYSES.

Permissible Variations in Mechanical Analysis.

4. The designated size for each grade together with the corresponding maximum permissible variations in mechanical analyses as determined by the use of laboratory screens and

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

with sieves as specified in the Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, except Fine Aggregates Used in Cement Concrete (Serial Designation: D 7) of the American Society for Testing Materials,[1] are given in the following table:

MAXIMUM PERMISSIBLE RANGE IN MECHANICAL ANALYSIS FOR EACH SIZE.
PERCENTAGE BY WEIGHT PASSING EACH SCREEN.

Designated Size, in.	Diameter of Screen Openings, in.							
	$\frac{1}{4}$	$\frac{1}{2}$	$\frac{3}{4}$	1	$1\frac{1}{2}$	2	$2\frac{1}{2}$	$3\frac{1}{2}$
0 – $\frac{1}{4}$ [a]	95–100	......	......	......	......	......	......	......
0 – $\frac{1}{4}$ [b]	85–100	......	......	......	......	......	......	......
$\frac{1}{4}$ – $\frac{1}{2}$	c	95–100	......	......	......	......	......	......
$\frac{1}{4}$ – $\frac{3}{4}$	c	......	95–100	......	......	......	......	......
$\frac{1}{4}$ – 1	c	25– 75	......	95–100	......	......	......	......
$\frac{1}{4}$ – $1\frac{1}{2}$	c	......	25– 75	......	95–100	......	......	......
$\frac{1}{4}$ – 2	c	......	......	25– 75	......	95–100	......	......
$\frac{1}{4}$ – $2\frac{1}{2}$	c	......	......	......	25– 75	......	95–100	......
1 – 2	......	......	......	0– 15	......	85–100	......	......
2 – $3\frac{1}{2}$	......	......	......	......	......	0– 15	......	85–100

[a] Additional requirements for grading shall be as follows:
Passing 20-mesh sieve.. 25 to 75 per cent
Passing 50-mesh sieve.. not over 25 "
Passing 100-mesh sieve.. " " 5 "

[b] Limits for silt and clay content may be inserted if desired.

[c] Any percentage from 0 to 10 per cent may be designated, with a maximum permissible variation therefrom of not more than $2\frac{1}{2}$ per cent.

IV. SIZE OF SAMPLES.

5. Samples of sand shall weigh at least 10 lb. Samples of gravel and samples composed of mixtures of sand and gravel shall weigh at least 50 times the weight of the largest piece therein. **Samples.**

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS
FOR
BROKEN STONE FOR WATERBOUND BASE[1]

Serial Designation: D 190 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

Material Covered.

1. These specifications cover the quality and grading of broken stone and stone screenings for use in a waterbound broken stone base course which is to be covered with any type of surface.

General Characteristics.

2. The broken stone shall consist of clean, tough, durable fragments, free from an excess of flat, elongated, soft or disintegrated pieces.

3. Stone for screenings shall be of suitable binding quality.

Physical Properties.

4. The stone shall conform to the following requirement:

Percentage of wear.................................not more than 7.0

$1\frac{1}{4}$ to $2\frac{1}{2}$ in. or $2\frac{1}{2}$ to $3\frac{1}{2}$ in. Size.

5. The course aggregate shall be of either (1) the $1\frac{1}{4}$ to $2\frac{1}{2}$-in. size or (2) the $2\frac{1}{2}$ to $3\frac{1}{2}$-in. size, and shall be well graded between the limits specified. When tested by means of laboratory screens, it shall conform to one of the following requirements:

(*a*)	Passing $1\frac{1}{4}$-in. screen........................	0 to 15 per cent
	Passing $2\frac{1}{2}$-in. screen........................	95 to 100 "
(*b*)	Passing $2\frac{1}{2}$-in. screen........................	0 to 15 per cent
	Passing $3\frac{1}{2}$-in. screen........................	95 to 100 "

NOTE.—Either of the above sizes or a combination thereof may be specified as desired.

0 to $\frac{1}{4}$-in. or 0 to $\frac{3}{4}$-in. Size.

6. The screenings shall be of either (1) the 0 to $\frac{1}{4}$-in. size, or (2) the 0 to $\frac{3}{4}$-in. size, well-graded from coarse to fine and free from dirt or other foreign material. When tested by means of laboratory screens they shall conform to one of the following requirements:

(*a*)	Passing $\frac{1}{4}$-in. screen........................	85 to 100 per cent
(*b*)	Passing $\frac{1}{4}$-in. screen........................	40 to 80 per cent
	Passing $\frac{3}{4}$-in. screen........................	95 to 100 "

NOTE.—Either of the above alternate sizes may be specified as desired.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

7. (*a*) The percentage of wear shall be determined in accordance with the Standard Method of Test for Abrasion of Road Material (Serial Designation: D 2) of the American Society for Testing Materials.[1] **Methods of Testing.**

(*b*) Determinations of size shall be made in accordance with the Standard Method of Mechanical Analysis of Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete (Serial Designation: D 18) of the American Society for Testing Materials.[1]

8. The methods of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[1] **Methods of Sampling.**

NOTE.—The test values for quality of stone incorporated in these specifications are suggested as the lowest safe requirements. It is not expected, however, that the limits for size and quality will be applicable to all localities and conditions.

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

BROKEN STONE FOR WATERBOUND MACADAM SURFACE COURSE[1]

Serial Designation: D 191 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

Material Covered. 1. These specifications cover the quality and grading of broken stone and broken stone screenings for use in waterbound macadam surface course.

General Characteristics. 2. The broken stone shall consist of clean, tough, durable fragments, free from an excess of flat, elongated, soft or disintegrated pieces.

3. Stone for screenings shall be of suitable binding quality.

Physical Properties. 4. The stone shall conform to the following requirements:

Percentage of wear	not more than 6
Toughness	not less than 6

$1\frac{1}{4}$ to $2\frac{1}{2}$ in. or $2\frac{1}{2}$ to $3\frac{1}{2}$-in. Size. 5. The coarse stone shall be of (1) the $1\frac{1}{4}$ to $2\frac{1}{2}$-in. size or (2) the $2\frac{1}{2}$ to $3\frac{1}{2}$-in size and shall be well graded between these limits. When tested by means of laboratory screens it shall conform to the following requirements:

(*a*)	Passing $1\frac{1}{4}$-in. screen	0 to 15 per cent
	Passing $2\frac{1}{2}$-in. screen	95 to 100 "
(*b*)	Passing $2\frac{1}{2}$-in. screen	0 to 15 per cent
	Passing $3\frac{1}{2}$-in. screen	95 to 100 "

NOTE.—Either of the above sizes or a combination thereof may be specified as desired.

0 to $\frac{1}{4}$-in. or 0 to $\frac{3}{4}$-in. Size. 6. The screenings shall be of either (1) the 0 to $\frac{1}{4}$-in. size, or (2) the 0 to $\frac{3}{4}$-in. size, well graded from coarse to fine and free from dirt or other foreign material. When tested by means of laboratory screens it shall conform to one of the following requirements:

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

(*a*)	Passing $\frac{1}{4}$-in. screen........................	85 to 100 per cent
(*b*)	Passing $\frac{1}{4}$-in. screen........................	40 to 80 per cent
	Passing $\frac{3}{4}$-in. screen........................	95 to 100 "

NOTE.—Either of the above alternate sizes may be specified as desired.

Methods of Testing.

7. (*a*) The percentage of wear shall be determined in accordance with the Standard Method of Test for Abrasion of Road Material (Serial Designation: D 2) of the American Society for Testing Materials.[1]

(*b*) The toughness shall be determined in accordance with the Standard Method of Test for Toughness of Rock (Serial Designation: D 3) of the American Society for Testing Materials.[1]

(*c*) Determinations of size shall be made in accordance with the Standard Method of Mechanical Analysis of Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete (Serial Designation: D 18) of the American Society for Testing Materials.[1]

Methods of Sampling.

8. The methods of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[1]

NOTE.—The test values for quality of stone incorporated in these specifications are suggested as the lowest safe requirements. It is not expected, however, that the limits for size and quality will be applicable to all localities and conditions.

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[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

BROKEN STONE FOR BITUMINOUS MACADAM[1]

Serial Designation: D 192 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

Material Covered. 1. These specifications cover the quality and grading of broken stone for use in a bituminous macadam surface course or bituminous macadam base.

General Characteristics. 2. The broken stone shall consist of clean, tough, durable fragments, free from an excess of flat, elongated, soft or disintegrated pieces, and free from stone coated with dirt or other objectionable matter.

Physical Properties. 3. The stone shall conform to the following requirements:

Percentage of wear	not more than 6
Toughness	not less than 6

$1\frac{1}{4}$ to $2\frac{1}{2}$-in. or $2\frac{1}{2}$ to $3\frac{1}{2}$-in. Size. 4. The coarse stone shall be either (1) the $1\frac{1}{4}$ to $2\frac{1}{2}$-in. size or (2) the $2\frac{1}{2}$ to $3\frac{1}{2}$-in. size and shall be well graded between the limits specified. When tested by means of laboratory screens it shall conform to one of the following requirements.

(*a*)	Passing $1\frac{1}{4}$-in. screen	0 to 15 per cent
	Passing $2\frac{1}{2}$-in. screen	95 to 100 "
(*b*)	Passing $2\frac{1}{2}$-in. screen	0 to 15 per cent
	Passing $3\frac{1}{2}$-in. screen	95 to 100 "

$\frac{3}{4}$ to $1\frac{1}{4}$-in. Size. 5. The stone for filling the surface voids in the coarse aggregate shall be the $\frac{3}{4}$ to $1\frac{1}{4}$-in. size, and when tested by means of laboratory screens shall conform to the following requirements:

Passing $\frac{3}{4}$-in. screen	0 to 15 per cent
Passing 1-in. screen	25 to 75 "
Passing $1\frac{1}{4}$-in. screen	95 to 100 "

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

6. Stone chips for use in the seal coat shall be of the $\frac{1}{4}$ to $\frac{3}{4}$-in. size, and when tested by means of laboratory screens shall conform to the following requirements: **$\frac{1}{4}$ to $\frac{3}{4}$-in. Size.**

Passing $\frac{1}{4}$-in. screen	0 to 15 per cent
Passing $\frac{1}{2}$-in. screen	25 to 75 "
Passing $\frac{3}{4}$-in. screen	95 to 100 "

7. (*a*) The percentage of wear shall be determined in accordance with the Standard Method of Test for Abrasion of Road Material (Serial Designation: D 2) of the American Society for Testing Materials.[1] **Methods of Testing.**

(*b*) The toughness shall be determined in accordance with the Standard Method of Test for Toughness of Rock (Serial Designation: D 3) of the American Society for Testing Materials.[1]

(*c*) Determinations of size shall be made in accordance with the Standard Method of Mechanical Analysis of Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete (Serial Designation: D 18) of the American Society for Testing Materials.[1]

8. The methods of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[1] **Methods of Sampling.**

NOTE.—The test values for quality of stone incorporated in these specifications are suggested as the lowest safe requirements. It is not expected, however, that the limits for size and quality will be applicable to all localities and conditions.

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS
FOR
BROKEN STONE FOR BITUMINOUS CONCRETE BASE[1]

Serial Designation: D 193 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

Material Covered. 1. These specifications cover the quality and grading of broken stone to be used in the construction of a closed mix and an open mix bituminous concrete base.

General Characteristics. 2. The broken stone shall consist of clean, tough, durable fragments, free from an excess of flat, elongated, soft or disintegrated pieces.

Physical Properties. 3. The stone shall conform to the following requirements:

Percentage of wear	not more than 6
Toughness	not less than 6

Coarse Aggregate for Open Mix Bituminous Concrete Base $1\frac{1}{4}$ to $2\frac{1}{2}$-in. Size 4. The coarse aggregate for open mix bituminous concrete base shall consist of the $1\frac{1}{4}$ to $2\frac{1}{2}$-in. size and shall be well graded between these limits. When tested by means of laboratory screens it shall conform to the following requirements:

Passing $1\frac{1}{4}$-in. screen	0 to 15 per cent
Passing $2\frac{1}{2}$-in. screen	95 to 100 "

Coarse Aggregate for Closed Mix Bituminous Concrete Base $\frac{1}{4}$ to $2\frac{1}{2}$ in. Size. 5. The coarse aggregate for closed mix bituminous concrete base shall consist of the $\frac{1}{4}$ to $2\frac{1}{2}$-in. size and shall be well graded between these limits. When tested by means of laboratory screens it shall conform to the following requirements:

Passing $\frac{1}{4}$-in. screen	0 to 5 per cent
Passing $1\frac{1}{4}$-in. screen	25 to 75 "
Passing $2\frac{1}{2}$-in. screen	95 to 100 "

Methods of Testing. 6. (*a*) The percentage of wear shall be determined in accordance with the Standard Method of Test for Abrasion of Road Material

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

(Serial Designation: D 2) of the American Society for Testing Materials.[1]

(*b*) The toughness shall be determined in accordance with the Standard Method of Test for Toughness of Rock (Serial Designation: D 3) of the American Society for Testing Materials.[1]

(*c*) Determinations of size shall be made in accordance with the Standard Method of Mechanical Analysis of Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete (Serial Designation: D 18) of the American Society for Testing Materials.[1]

Methods of Sampling.

7. The methods of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[1]

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

BROKEN STONE FOR BITUMINOUS CONCRETE SURFACE[1]

Serial Designation: D 194 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

Material Covered.

1. These specifications cover the quality and grading of broken stone to be used in the construction of fine-graded bituminous concrete pavements (Modified Topeka, or stone-filled sheet asphalt) and coarse-graded bituminous concrete, including asphaltic concrete binder.

General Characteristics.

2. The broken stone shall consist of clean tough durable fragments free from an excess of flat, elongated, soft, or disintegrated pieces, and free from stone coated with dirt or other objectionable matter.

Physical Properties.

3. The stone shall conform to the following requirements:

Percentage of wear	not more than 6
Toughness	not less than 6

Stone Chips for Seal Coat ¼ to ½-in. Size.

4. Stone chips shall be of the ¼ to ½-in. size. When tested by means of laboratory screens they shall conform to the following requirements:

Passing ¼-in. screen	0 to 15 per cent
Passing ½-in. screen	95 to 100 "

Coarse Aggregate for Fine-graded Bituminous Concrete Mixture ¼ to ¾-in. Size.

5. The coarse aggregate for fine-grade bituminous concrete shall be of the ¼ to ¾-in. size and shall be well graded between these limits. When tested by means of laboratory screens it shall conform to the following requirements:

Passing ¼-in. screen	0 to 15 per cent
Passing ½-in. screen	25 to 75 "
Passing ¾-in. screen	95 to 100 "

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

Coarse Aggregate for Coarse-graded Bituminous Concrete Mixture ¼ to 1¼-in. Size.

6. The coarse aggregate for coarse-graded bituminous concrete shall consist of the ¼ to 1¼-in. size and shall be well graded between these limits. When tested by means of laboratory screens it shall conform to the following requirements:

Passing ¼-in. screen	0 to 10 per cent
Passing ¾-in. screen	25 to 75 "
Passing 1½-in. screen	95 to 100 "

Methods of Testing.

7. (*a*) The percentage of wear shall be determined in accordance with the Standard Method of Test for Abrasion of Road Material (Serial Designation: D 2) of the American Society for Testing Materials.[1]

(*b*) The toughness shall be determined in accordance with the Standard Method of Test for Toughness of Rock (Serial Designation: D 3) of the American Society for Testing Materials.[1]

(*c*) Determinations of size shall be made in accordance with the Standard Method of Mechanical Analysis of Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete (Serial Designation: D 18) of the American Society for Testing Materials.[1]

Methods of Sampling.

8. The methods of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[1]

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

BROKEN SLAG FOR BITUMINOUS MACADAM BASE[1]

Serial Designation: D 195 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

Material Covered.

1. These specifications cover $\frac{3}{4}$ to $1\frac{1}{4}$-in., $1\frac{1}{4}$ to $2\frac{1}{2}$-in., and $2\frac{1}{2}$ to $3\frac{1}{2}$-in. size broken slag to be used in the construction of a bituminous macadam base in which the bituminous material is applied by the penetration method and which is to be covered with any type of surface course.

The $2\frac{1}{2}$ to $3\frac{1}{2}$-in. size is intended for use as a first course, which will be penetrated with bituminous material, when a base of over $3\frac{1}{2}$ in. is desired.

The $1\frac{1}{4}$ to $2\frac{1}{2}$-in. size is intended for use as a second course which is to be penetrated with bituminous material.

The $\frac{3}{4}$ to $1\frac{1}{4}$-in. size is intended to fill the surface voids when a sheet asphalt or a fine-graded bituminous concrete is to be placed directly upon it. This material can be omitted when a coarse-graded bituminous concrete or a binder course is to be laid directly upon the base.

General Characteristics.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot of each size specified shall be not less than 65 lb.

$2\frac{1}{2}$ to $3\frac{1}{2}$-in. and $1\frac{1}{4}$ to $2\frac{1}{2}$-in. Size.

4. The coarse slag when tested by means of laboratory screens shall conform to the following requirements:

(*a*) Passing $3\frac{1}{2}$-in. screen.................. not less than 95 per cent
Total passing $2\frac{1}{2}$-in. screen............ not more than 15 "

(*b*) Passing $2\frac{1}{2}$-in. screen.................. not less than 95 per cent
Total passing $1\frac{1}{4}$-in. screen............ not more than 15 "

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

5. The intermediate size slag when tested by means of laboratory screens shall conform to the following requirements: **$\frac{3}{4}$ to $1\frac{1}{4}$-in. Size.**

Passing $1\frac{1}{4}$-in. screen	not less than 95 per cent
Passing 1-in. screen	25 to 75 "
Passing $\frac{3}{4}$-in. screen	not more than 15 "

6. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[1] **Methods of Testing.**

7. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[1] **Sampling.**

NOTE.—The test values for quality of slag incorporated in these specifications are suggested as the lowest safe requirements. It is not expected, however, that the limits for size and quality will be applicable to all conditions.

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

BROKEN SLAG FOR BITUMINOUS CONCRETE BASE[1]

Serial Designation: D 196 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

Material Covered. 1. These specifications cover $\frac{1}{4}$ to $1\frac{1}{4}$-in. and $\frac{1}{4}$ to $2\frac{1}{2}$-in. size broken slag, either of which is to be used in the construction of a bituminous concrete base prepared by the mixing method and upon which may be placed any type of surface course.

General Characteristics. 2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight. 3. The weight per cubic foot of each size specified shall be not less than 70 lb.

$\frac{1}{4}$ to $1\frac{1}{4}$-in. or $\frac{1}{4}$ to $2\frac{1}{2}$-in. Size. 4. The slag when tested by means of laboratory screens shall conform to one of the following requirements:

(*a*)	Passing $1\frac{1}{4}$-in. screen	not less than 95 per cent
	Passing $\frac{3}{4}$-in. screen	25 to 75 "
	Passing $\frac{1}{4}$-in. screen	not more than 5 "
(*b*)	Passing $2\frac{1}{2}$-in. screen	not less than 95 per cent
	Passing $1\frac{1}{4}$-in. screen	25 to 75 "
	Passing $\frac{1}{4}$-in. screen	not more than 5 "

NOTE.—One of the above alternate sizes for broken slag should be specified to best suit the local and service conditions.

Methods of Testing. 5. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[2]

Methods of Sampling. 6. The methods of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[2]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

BROKEN SLAG FOR BITUMINOUS MACADAM WEARING COURSE[1]

Serial Designation: D 159 - 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1924.

Material Covered.

1. These specifications cover $\frac{3}{4}$ to $\frac{1}{4}$-in., $1\frac{1}{4}$ to $\frac{3}{4}$-in., and $2\frac{1}{2}$ to $1\frac{1}{4}$-in. or $3\frac{1}{2}$ to $2\frac{1}{2}$-in. size slag to be used in the construction of a bituminous macadam wearing course in which the bituminous material is applied by the penetration method. The maximum size of $3\frac{1}{2}$ in. specified for the wearing course is on the basis of a course $2\frac{1}{2}$ to $3\frac{1}{2}$ in. in thickness after consolidation. The $1\frac{1}{4}$ to $\frac{3}{4}$-in. size should be used to fill the surface voids after the first application of bituminous material, and the $\frac{3}{4}$ to $\frac{1}{4}$-in. size after the application of the seal coat.

General Characteristics of Slag.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot of each size specified shall be not less than 70 lb.

¾ to ¼-in. Size.

4. The $\frac{3}{4}$ to $\frac{1}{4}$-in. size is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall meet the following requirements:

Passing $\frac{3}{4}$-in. screen	not less than 95 per cent
Passing $\frac{1}{2}$-in. screen	25 to 75 "
Passing $\frac{1}{4}$-in. screen	not more than 15 "

1¼ to ¾-in. Size.

5. The $1\frac{1}{4}$ to $\frac{3}{4}$-in. size is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall meet the following requirements.

Passing $1\frac{1}{4}$-in. screen	not less than 95 per cent
Passing 1-in. screen	25 to 75 "
Passing $\frac{3}{4}$-in. screen	not more than 15 "

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

2½ to 1¾-in. Size. 6. The $2\frac{1}{2}$ to $1\frac{1}{4}$-in. and $3\frac{1}{2}$ to $2\frac{1}{2}$-in. sizes are that portion of the product of the crusher, which, when tested by means of laboratory screens, shall conform to one of the following requirements:

Passing $2\frac{1}{2}$-in. screen	not less than 95 per cent
Total passing $1\frac{1}{4}$-in. screen	not more than 15 "
Passing $3\frac{1}{2}$-in. screen	not less than 95 per cent
Total passing $2\frac{1}{2}$-in. screen	not more than 15 "

NOTE.—One of the above alternate sizes for coarse slag should be specified to best suit the local and service conditions.

Methods of Testing. 7. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[1]

Sampling. 8. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[1]

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

BROKEN SLAG FOR BITUMINOUS CONCRETE (COARSE-GRADED AGGREGATE TYPE)[1]

Serial Designation: D 160 - 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

Issued, 1923; Revised, 1924.

Material Covered. 1. These specifications cover ¾ to ¼-in. and 1¼ to ¼-in. size slag to be used in the construction of a coarse-graded bituminous concrete wearing course constructed by the mixing method. The ¾ to ¼-in. size should be used after the application of the seal coat when a seal coat is used.

General Characteristics of Slag. 2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight. 3. The weight per cubic foot of each size specified shall be not less than 70 lb.

¾ to ¼-in. Size. 4. The ¾ to ¼-in. size is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall meet the following requirements:

Passing ¾-in. screen	not less than 95 per cent
Passing ½-in. screen	25 to 75 "
Passing ¼-in. screen	not more than 15 "

1¼ to ¼-in. Size. 5. The 1¼ to ¼-in. size is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall meet the following requirements:

Passing 1¼-in. screen	95 to 100 per cent
Passing ¾-in. screen	25 to 75 "
Passing ¼-in. screen	not more than 5 "

Methods of Testing. 6. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[1]

Sampling. 7. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[1]

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

BROKEN SLAG FOR BITUMINOUS CONCRETE (FINE-GRADED AGGREGATE TYPE)[1]

Serial Designation: D 161 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1924.

Material Covered.

1. These specifications cover slag aggregate to be used in the construction of a bituminous concrete wearing surface constructed by the mixing method.

General Characteristics of Slag.

2. The broken slag shall be air-cooled blast-furnace slag and shall consist of angular fragments reasonably uniform in density and quality, and reasonably free from thin, elongated or glassy pieces, dirt or other objectionable matter.

Weight.

3. The weight per cubic foot shall be not less than 70 lb.

Slag Aggregate 0 to ½-in. Size.

4. The slag aggregate is that portion of the product of the crusher, which, when tested by means of laboratory screens, shall conform to the following requirements:

Passing ½-in. screen	95 to 100 per cent
Passing ¼-in. screen	0 to 75 "

Methods of Testing.

5. *Weight per Cubic Foot.*—The weight per cubic foot shall be determined in accordance with the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29) of the American Society for Testing Materials.[2]

Sampling.

6. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[2]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS
FOR
SAND FOR SHEET ASPHALT AND BITUMINOUS CONCRETE PAVEMENTS[1]

Serial Designation: D 162 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923

Material Covered. 1. These specifications cover sand for use in the construction of the surface course of sheet asphalt pavements and those types of bituminous concrete pavements in which sand predominates.

Physical Properties. 2. The grains of the sand shall be clean, tough, rough surfaced and angular. The sand after drying shall be free from lumps or balls of clay or of clay and sand. Before being incorporated into the paving mixture it shall conform to the following requirements as to grading:

Passing 200-mesh sieve	0 to 5 per cent	
Passing 100-mesh sieve, retained on 200-mesh	6 to 25 "	15 to 40 per cent
Passing 80-mesh sieve, retained on 100-mesh	6 to 25 "	
Passing 50-mesh sieve, retained on 80-mesh	5 to 40 "	30 to 60 "
Passing 40-mesh sieve, retained on 50-mesh	5 to 30 "	
Passing 30-mesh sieve, retained on 40-mesh	8 to 25 "	18 to 50 "
Passing 20-mesh sieve, retained on 30-mesh	5 to 15 "	
Passing 10-mesh sieve, retained on 20-mesh	5 to 15 "	
Passing 10-mesh sieve	95 to 100 "	
Passing ¼-in. screen	100 "	

Methods of Testing. 3. *General Characteristics.*—The general characteristics shall be determined by examination under a microscope or a magnifying glass.

4. *Mesh Composition.*—The tests for the mechanical analysis shall be made in accordance with the Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, Except Fine Aggregates Used in Cement Concrete (Serial Designation: D 7) of the American Society for Testing Materials.[2]

Sampling. 5. The method of sampling shall conform to the Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (Serial Designation: D 75) of the American Society for Testing Materials.[2]

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

NATURAL OR ARTIFICIAL SAND-CLAY MIXTURES FOR ROAD SURFACING[1]

Serial Designation: D 67 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923

General. 1. The sand-clay shall be composed of either a naturally occurring or artificially prepared mixture of hard, durable, preferably angular, fragments of sand together with silt and clay with or without gravel, and shall be free from an excess of feldspar or mica.

Grading. 2. (*a*) When tested by means of laboratory sieves and screens the material shall conform to the following requirements as to grading:

Passing 2-in. screen	100 per cent
Passing $\frac{1}{4}$-in. screen	30 to 100 "

(*b*) The material, if any, retained on the $\frac{1}{4}$-in. screen shall be uniformly graded from the maximum size present to $\frac{1}{4}$ in.

(*c*) The material passing the $\frac{1}{4}$-in. screen shall conform to the following requirements:

Total sand	50 to 80 per cent
Sand retained on 60-mesh	30 to 60 "
Silt	5 to 20 "
Clay	15 to 30 "

Methods of Testing. 3. The tests for the mechanical analysis of the sand, clay, natural sand-clay or topsoil mixtures shall be made in accordance with the Tentative Method of Mechanical Analysis of Subgrade Soils (Serial Designation: D 137 – 25 T) of the American Society for Testing Materials,[2] except that the determination of the percentage of suspension clay may be omitted and the percentage of total clay may be calculated by difference as follows:

$$100 - (\text{Percentage of Sand} + \text{Percentage of Silt}) = \text{Percentage of Clay.}$$

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] See p. 490.

TENTATIVE SPECIFICATIONS

FOR

CALCIUM CHLORIDE FOR DUST PREVENTION.[1]

Serial Designation: D 98 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1922.

Material Covered.

1. These specifications cover calcium chloride to be applied to the surface of highways as a dust preventive.

I. PROPERTIES AND TESTS.

Physical Properties.

2. The calcium chloride shall be in the form of loose dry lumps or flakes, and fine enough to feed readily through the common forms of spreaders used in road work, and when tested by means of laboratory screens and sieves shall meet the following requirements:

Passing $\frac{3}{8}$-in. screen	100 per cent
Retained on $\frac{1}{4}$-in. screen	not more than 20 "
Passing 20-mesh sieve	not more than 10 "

Chemical Composition.

3. Ten grams of the material dissolved in 100 cc. of boiling water shall show less than 1.0 per cent insoluble residue. Not more than 1.0 cc. of normal acid shall be required to neutralize the alkalinity of the filtrate, using methyl orange. The chemical composition shall conform to the following requirements:

$CaCl_2$ (anhydrous)	not less than 73. per cent
$MgCL_2$	not more than 0.5 "
NaCl	not more than 2.0 "
Other impurities	not more than 1.0 "

The percentage of calcium chloride shall be calculated from the percentage of calcium in the filtrate above referred to, after making a deduction for the SO_4, which shall be assumed to be in combination as $CaSO_4$.

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

II. PACKAGES.

4. The calcium chloride shall be delivered in air-tight moisture-proof bags or sacks containing approximately 100 lb. each, or in air-tight steel drums weighing not more than 350 lb. each. The name of the manufacturer, the lot number, the approximate net weight, and the percentages of calcium chloride guaranteed by the manufacturer shall be plainly marked on each container. **Packages.**

III. INSPECTION.

5. Every facility shall be provided the purchaser should he elect to have his own representative sample the material at the plant. If the purchaser decides to sample the material after delivery in the field it is understood that a 3-per-cent variation in content of $CaCl_2$ from the chemical composition stated in Section 3 shall be permissible. **Inspection.**

6. Calcium chloride shall be rejected if it fails to pass any of the requirements of these specifications and if it has become caked or sticky in the containers before opening. **Rejection.**

IV. BASIS OF PAYMENT.

7. Payment will be made on the basis of total number of pounds of 100-per-cent calcium chloride delivered. **Basis of Payment.**

TENTATIVE SPECIFICATIONS
FOR
ASPHALT CEMENT, 10 TO 15 PENETRATION, FOR THE MANUFACTURE OF ASPHALT BLOCK.[1]

Serial Designation: D 133 - 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923.

Properties 1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec.......10 to 15

(*b*) Flash Point (open cup).....not less than 200° C. (392° F.)

(*c*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr............ ....not more than 1 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 160° C. (325° F.) as compared with penetration of asphalt cement before heating, not less than 50 per cent

(*d*) Ductility at 25° C. (77[1] F.)................5 cm. to 15 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride....... ....not less than 99 per cent

NOTE.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide) shall be reported.

Methods of Testing. 2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5).[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92).[3]

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6).[3]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T).[4]

(*e*) *Proportion of Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165 – 23 T).[5]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted in 1925.

[3] 1924 Book of A.S.T.M. Standards.

[4] See p. 501. [5] See p. 499.

TENTATIVE SPECIFICATIONS
FOR
ASPHALT CEMENT, 15 TO 25 PENETRATION, FOR THE MANUFACTURE OF ASPHALT BLOCK.[1]

Serial Designation: D 134 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923.

Properties.

1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec.........15 to 25

(*b*) Flash Point (Open Cup)......not less than 200° C. (392° F.)

(*c*) Loss on Heating at 163° C. (325° F.), 50 g., 5 hr.......... ....not more than 1 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt cement before heating, not less than 50 per cent

(*d*) Ductility at 25° C. (77° F.)................5 cm. to 20 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride....... ....not less than 99 per cent

NOTE.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide) shall be reported.

Methods of Testing.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5).[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92).[3]

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6).[3]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T).[4]

(*e*) *Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165 – 23 T).[5]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted in 1925.

[3] 1924 Book of A.S.T.M. Standards.

[4] See p. 501. [5] See p. 499.

TENTATIVE SPECIFICATIONS

FOR

ASPHALT CEMENT, 25 TO 30 PENETRATION, FOR USE IN SHEET ASPHALT AND ASPHALTIC-CONCRETE PAVEMENTS[1]

Serial Designation: D 163 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923

Properties. 1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec........25 to 30

(*b*) Flash Point (open cup).......not less than 175° C. (347° F.)

(*c*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr.........................not more than 2 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt cement before heating, not less than 60 per cent

(*d*) Ductility at 25° C. (77° F.)..............not less than 15 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride...................not less than 99 per cent

NOTE.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide) shall be reported.

Methods of Testing. 2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5).[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92).[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted in 1925.

[3] 1924 Book of A.S.T.M. Standards.

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6).[1]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T).[2]

(*e*) *Proportion of Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165 – 23 T).[3]

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[1] 1924 Book of A.S.T.M. Standards.

[2] See p. 501.

[3] See p. 499.

TENTATIVE SPECIFICATIONS

FOR

ASPHALT CEMENT, 30 TO 40 PENETRATION, FOR USE IN SHEET ASPHALT AND ASPHALTIC-CONCRETE PAVEMENTS[1]

Serial Designation: D 164 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923

Properties. 1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec. . . . . .30 to 40

(*b*) Flash Point (open cup) . . . . . .not less than 175° C. (347° F.)

(*c*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr. . . . . .not more than 2 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt cement before heating, not less than 60 per cent

(*d*) Ductility at 25° C. (77° F.) . . . . .not less than 25 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride . . . . .not less than 99 per cent

NOTE.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide) shall be reported.

Methods of Testing. 2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5).[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92).

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted in 1925.

[3] 1924 Book of A.S.T.M. Standards.

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6).[1]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T).[2]

(*e*) *Proportion of Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165 – 23 T).[3]

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[1] 1924 Book of A.S.T.M. Standards.

[2] See p. 501.

[3] See p. 499.

TENTATIVE SPECIFICATIONS

FOR

ASPHALT CEMENT, 40 TO 50 PENETRATION, FOR USE IN SHEET ASPHALT AND ASPHALTIC-CONCRETE PAVEMENTS.[1]

Serial Designation: D 99 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1922, 1923.

Properties. 1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec. . . . . .40 to 50

(*b*) Flash point (open cup). . . . . . . not less than 175° C. (347° F.)

(*c*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr. . . . . not more than 2 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt cement before heating. . not less than 60 per cent

(*d*) Ductility at 25° C. (77° F.). . . . . not less than 30 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride. . . . . not less than 99 per cent

NOTE.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide) shall be reported.

Methods of Testing. 2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5);[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92);[3]

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6);[3]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T);[4]

(*e*) *Proportion of Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165 – 23 T).[5]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted in 1925.

[3] 1924 Book of A.S.T.M. Standards. [4] See p. 501. [5] See p. 499.

TENTATIVE SPECIFICATIONS
FOR
ASPHALT CEMENT, 50 TO 60 PENETRATION, FOR USE IN SHEET ASPHALT AND ASPHALTIC-CONCRETE PAVEMENTS.[1]

Serial Designation: D 100 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1922, 1923.

Properties.

1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec......50 to 60

(*b*) Flash point (open cup)........not less than 175° C. (347° F.)

(*c*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr.......................................not more than 2 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt cement before heating. .not less than 60 per cent

(*d*) Ductility at 25° C. (77° F.)............not less than 30 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride...................................not less than 99 per cent

Note.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide) shall be reported.

Methods of Testing.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5);[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92);[3]

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6);[3]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T);[4]

(*e*) *Proportion of Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165 – 23 T).[5]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before Jannary 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted in 1925.

[3] 1924 Book of A.S.T.M. Standards. [4] See p. 501. [5] See p. 499.

TENTATIVE SPECIFICATIONS

FOR

ASPHALT CEMENT, 60 TO 70 PENETRATION, FOR USE IN SHEET ASPHALT, ASPHALTIC CONCRETE, AND ASPHALT-MACADAM PAVEMENTS.[1]

Serial Designation: D 101 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1922, 1923.

Properties. 1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec.......60 to 70

(*b*) Flash point (open cup)........not less than 175° C. (347° F.)

(*c*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr...not more than 2 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt cement before heating..not less than 60 per cent

(*d*) Ductility at 25° C. (77° F.)...........not less than 30 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride...................................not less than 99 per cent

NOTE.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide) shall be reported.

Methods of Testing. 2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5);[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92);[3]

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6);[3]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T);[4]

(*e*) *Proportion of Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165 – 23 T).[5]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted in 1925.

[3] 1924 Book of A.S.T.M. Standards. [4] See p. 501. [5] See p. 499.

TENTATIVE SPECIFICATIONS
FOR
ASPHALT CEMENT, 85 TO 100 PENETRATION, FOR USE IN ASPHALT-MACADAM PAVEMENTS.[1]

Serial Designation: D 102 – 24 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1922, 1923, 1924.

Properties.

1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec. . . . . . . 85 to 100

(*b*) Flash point (open cup) . . . . not less than 175° C. (347° F.)

(*c*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr. . . . . not more than 2 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt cement before heating . . not less than 60 per cent

(*d*) Ductility at 25° C. (77° F.) . . . . not less than 30 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride . . . . not less than 99 per cent

Note.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide) shall be reported.

Methods of Testing.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5);[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92);[3]

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6);[3]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T);[4]

(*e*) *Proportion of Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165 – 23 T).[5]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted 1925.

[3] 1924 Book of A.S.T.M. Standards. [4] See p. 501. [5] See p. 499.

TENTATIVE SPECIFICATIONS

FOR

ASPHALT CEMENT, 100 TO 120 PENETRATION, FOR USE IN ASPHALT-MACADAM PAVEMENTS.[1]

Serial Designation: D 103 – 24 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1922, 1923, 1924.

Properties. 1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec.....100 to 120

(*b*) Flash point (open-cup)........not less than 175° C. (347° F.)

(*c*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr...........
..............................not more than 2 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt cement before heating..not less than 60 per cent

(*d*) Ductility at 25° C. (77° F.)............not less than 30 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride.......
.............................not less than 99 per cent

NOTE.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide shall be reported.

Methods of Testing. 2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5);[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92);[3]

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6);[3]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T);[4]

(*e*) *Proportion of Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165 – 23 T).[5]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted in 1925.

[3] 1924 Book of A.S.T.M. Standards. [4] See p. 501. [5] See p. 499.

TENTATIVE SPECIFICATIONS

FOR

ASPHALT CEMENT, 120 TO 150 PENETRATION, FOR USE IN ASPHALT-MACADAM PAVEMENTS.[1]

Serial Designation: D 135-23 T.

This is a Tentative Standard only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923.

Properties.

1. The asphalt cement shall be homogeneous and free from water. It shall conform to the following requirements:

(*a*) Penetration at 25° C. (77° F.), 100 g., 5 sec. 120 to150

(*b*) Flash point (open cup). . . . . .not less than 175° C. (347° F.)

(*c*) Loss on heating at 163° C. (325° F.), 50 g., 5 hr. . . . . .not more than 2 per cent

Penetration at 25° C. (77° F.), 100 g., 5 sec., of residue after heating at 163° C. (325° F.) as compared with penetration of asphalt cement before heating. .not less than 60 per cent

(*d*) Ductility at 25° C. (77° F.). . . . . .not less than 30 cm.

(*e*) Proportion of bitumen soluble in carbon tetrachloride. . . . . .not less than 99 per cent

NOTE.—When less than 99 per cent of the asphalt cement is soluble in carbon tetrachloride, the percentage of bitumen (solubility in carbon disulfide) shall be reported.

Methods of Testing.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Penetration:* Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5).[2]

(*b*) *Flash Point:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92).[3]

(*c*) *Loss on Heating:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6).[3]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113-22 T.).[4]

(*e*) *Proportion of Bitumen Soluble in Carbon Tetrachloride:* Tentative Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (Serial Designation: D 165-23 T).[5]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] A.S.T.M. Standards Adopted in 1925.

[3] 1924 Book of A.S.T.M. Standards. [4] See p. 501. [5] See p. 499.

TENTATIVE SPECIFICATIONS
FOR
HIGH-CARBON TAR FOR SURFACE TREATMENT, COLD APPLICATION.[1]

Serial Designation: D 104 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1923.

Properties.

1. The tar shall conform to the following requirements:[2]

(*a*) Water........................not more than 2.00 per cent

(*b*) Specific viscosity, Engler,[3] 50 cc. at 40° C. (104° F.)..8 to 35

(*c*) Distillation test on water-free material:

Total Distillate, by weight, 0 to 170° C. (32 to 338° F.)..................	not more than 7.00 per cent
Total Distillate, by weight, 0 to 235° C. (32 to 455° F.)..................	not more than 20.00 "
Total Distillate, by weight, 0 to 270° C. (32 to 518° F.)..................	not more than 30.00 "
Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)..................	not more than 35.00 "
Residue, by weight	not less than 65.00 "

(*d*) Specific gravity at 25°/25° C. (77°/77° F.) of total distillate to 300° C. (572° F.) ....not less than 1.01

(*e*) Softening point (Ring-and-Ball Method) of residue from distillation test.......................not more than 60° C. (140° F.)

(*f*) Total Bitumen (Soluble in Carbon Disulphide)..88 to 97 per cent

Methods of Testing.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials, except as specified in Paragraph (*b*):

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] Within the viscosity limits designated a material should be chosen to meet the local conditions of temperature, road conditions and climate. It is recommended that materials be called for under the following range and headings: Light 8–13, Medium 13–18, Heavy 18–25, Extra Heavy 25–35.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[1]

(*b*) *Specific Viscosity:*[2] Method contained in *Bulletin No. 691*, U. S. Department of Agriculture, the instrument being standardized by the Bureau of Standards. The results shall be reported as specific viscosity compared with water at 25° C. (77° F.).

(*c*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20);[1]

(*d*) *Softening Point:* Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36);[1]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 – 23 T).[3]

[1] 1924 Book of A.S.T.M. Standards.

[2] The Engler test has not been standardized by the American Society for Testing Materials. Committees of the Society have under consideration the Saybolt Furol Apparatus, which may be adopted later.

[3] See p. 496.

TENTATIVE SPECIFICATIONS

FOR

LOW-CARBON TAR FOR SURFACE TREATMENT, COLD APPLICATION[1]

Serial Designation: D 105 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1923.

Properties. 1. The tar shall conform to the following requirements:[2]

(*a*) Water.........................not more than 2.00 per cent

(*b*) Specific viscosity, Engler,[3] 50 cc. at 40° C. (104° F.)...8 to 35

(*c*) Distillation test on water-free material:

Total Distillate, by weight, 0 to 170° C. (32 to 338° F.)	not more than 5.00 per cent
Total Distillate, by weight, 0 to 235° C. (32 to 455° F.)	not more than 20.00 "
Total Distillate, by weight, 0 to 270° C. (32 to 518° F.)	not more than 35.00 "
Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)	not more than 45.00 "
Residue, by weight	not less than 55.00 "

(*d*) Softening point (Ring-and-Ball Method) of residue from distillation test.........................not more than 60° C. (140° F.)

(*e*) Total Bitumen (Soluble in Carbon Disulfide)...............
..........................not less than 95.00 per cent

Methods of Testing. 2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials, except as specified in Paragraph (*b*):

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] Within the viscosity limits designated a material should be chosen to meet the local conditions of temperature, road conditions and climate. It is recommended that materials be called for under the following range and headings: Light 8–13, Medium 13–18, Heavy 18–25, Extra Heavy 25–35.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[1]

(*b*) *Specific Viscosity:*[2] Method contained in *Bulletin No. 691*, U. S. Department of Agriculture, the instrument being standardized by the Bureau of Standards. The results shall be reported as specific viscosity compared with water at 25° C. (77° F.).

(*c*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20);[1]

(*d*) *Softening Point:* Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36);[2]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 – 23 T).[3]

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[1] 1924 Book of A.S.T.M. Standards.

[2] The Engler test has not been standardized by the American Society for Testing Materials. Committees of the Society have under consideration the Saybolt Furol apparatus, which may be adopted later.

[3] See p. 496.

TENTATIVE SPECIFICATIONS

FOR

HIGH-CARBON TAR CEMENT FOR USE COLD IN REPAIR WORK (CUT-BACK PRODUCT).[1]

Serial Designation: D 106 – 25 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1923, 1925.

Properties

1. The tar cement shall conform to the following requirements:[2]

(*a*) Water........................not more than 2.00 per cent

(*b*) Specific viscosity, Engler,[3] 50 cc. at 40° C. (104° F.)..35 to 80

(*c*) Distillation test on water-free material:

Total Distillate, by weight, 0 to 170° C. (32 to 338° F.)...................	2.00 to 8.00	per cent
Total Distillate, by weight, 0 to 235° C. (32 to 455° F.)...................	8.00 to 20.00	"
Total Distillate, by weight, 0 to 270° C. (32 to 518° F.)...................	18.00 to 30.00	"
Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)...................	not more than 35.00	"
Residue, by weight...................	not less than 65.00	"

(*d*) Softening point (Ring-and-Ball Method) of residue from distillation test........................not more than 65° C. (149° F.)

(*e*) Total Bitumen (Soluble in Carbon Disulfide)[4]..78 to 95 per cent

Methods of Testing.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials, except as specified in Paragraph (*b*):

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] It is recommended that materials be called for under the following range and headings: Light 35 to 60, Heavy 60 to 80. The heavy material may require heating before use and care should be taken to avoid foaming on account of the possible water content.

[4] The specification range for total bitumen covers a wide variety of materials. If products from vertical retort or low-carbon coke-oven tars are desired, a range of 88 to 95 per cent should be specified. If high-carbon coke-oven or mixtures of coke-oven and gas-house tars are desired, a range of 78 to 88 per cent should be specified.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[1]

(*b*) *Specific Viscosity:*[2] Method contained in *Bulletin No. 691*, U. S. Department of Agriculture, the instrument being standardized by the Bureau of Standards. The results shall be reported as specific viscosity compared with water at 25° C. (77° F.).

(*c*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20);[1]

(*d*) *Softening Point:* Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36);[1]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 – 23 T).[3]

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[1] 1924 Book of A.S.T.M. Standards.

[2] The Engler test has not been standardized by the American Society for Testing Materials. Committees of the Society have under consideration the Saybolt Furol apparatus, which may be adopted later.

[3] See p. 496.

TENTATIVE SPECIFICATIONS

FOR

LOW-CARBON TAR CEMENT FOR USE COLD IN REPAIR WORK (CUT-BACK PRODUCT).[1]

Serial Designation: D 107 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1923.

Properties.

1. The tar cement shall conform to the following requirements:[2]

(*a*) Water........................not more than 2.00 per cent

(*b*) Specific viscosity, Engler,[3] 50 cc. at 40° C. (104° F.)..35 to 80

(*c*) Distillation test on water-free material:

Total Distillate, by weight, 0 to 170° C. (32 to 338° F.)..................	3.00 to 10.00 per cent.
Total Distillate, by weight, 0 to 235° C. (32 to 455° F.)..................	8.00 to 20.00 "
Total Distillate, by weight, 0 to 270° C. (32 to 518° F.)..................	18.00 to 30.00 "
Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)..................	not more than 38.00 "
Residue, by weight..................	not less than 62.00 "

(*d*) Softening point (Ring-and-Ball Method) of residue from distillation test.......................not more than 65° C. (149° F.)

(*e*) Total Bitumen (Soluble in Carbon Disulfide)..not less than 95 per cent

Methods of Testing.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials, except as specified in Paragraph (*b*):

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] It is recommended that materials be called for under the following range and headings: Light 35 to 60, Heavy 60 to 80. The heavy material may require heating before use and care should be taken to avoid foaming on account of the possible water content.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[1]

(*b*) *Specific Viscosity:*[2] Method contained in *Bulletin No. 691*, U. S. Department of Agriculture, the instrument being standardized by the Bureau of Standards. The results shall be reported as specific viscosity compared with water at 25° C. (77° F.).

(*c*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20);[1]

(*d*) *Softening Point:* Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36);[1]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 – 23 T).[3]

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[1] 1924 Book of A.S.T.M. Standards.

[2] The Engler test has not been standardized by the American Society for Testing Materials. Committees of the Society have under consideration the Saybolt Furol apparatus, which may be adopted later.

[3] See p. 496.

TENTATIVE SPECIFICATIONS

FOR

HIGH-CARBON TAR FOR SURFACE TREATMENT, HOT APPLICATION.[1]

Serial Designation: D 108 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1923.

Properties

1. The tar shall conform to the following requirements:[2]

(*a*) Water .... 0.00 per cent

(*b*) Float test at 32° C. (89.6° F.) .... 60 to 150 sec.

(*c*) Distillation test:

Total Distillate, by weight, 0 to 170° C. (32 to 338° F.)	not more than	1.00 per cent
Total Distillate, by weight, 0 to 235° C. (32 to 455° F.)	not more than 10.00	"
Total Distillate, by weight, 0 to 270° C. (32 to 518° F.)	not more than 15.00	"
Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)	not more than 25.00	"
Residue, by weight	not less than 75.00	"

(*d*) Specific gravity at 25° C. (77° F.) of total distillate to 300° C. (572° F.) .... not less than 1.03

(*e*) Softening point (Ring-and-Ball Method) of residue from distillation test .... not more than 65° C. (149° F.)

(*f*) Total Bitumen (Soluble in Carbon Disulfide)[3] .... 78 to 95 per cent

Methods of Testing.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] The specification range for total bitumen covers a wide variety of materials. If products from vertical retort or low-carbon coke-oven tars are desired, a range of 88 to 95 per cent should be specified. If high-carbon coke-oven or mixtures of coke-oven and gas-house tars are desired, a range of 78 to 88 per cent should be specified.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[1]

(*b*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20);[1]

(*c*) *Softening Point:* Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36);[1]

(*d*) *Float Test:* Tentative Method of Float Test for Bituminous Materials (Serial Designation: D 139 – 25 T);[2]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 – 23 T).[3]

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[1] 1924 Book of A.S.T.M. Standards.

[2] See p. 503.

[3] See p. 496.

TENTATIVE SPECIFICATIONS

FOR

LOW-CARBON TAR FOR SURFACE TREATMENT, HOT APPLICATION.[1]

Serial Designation: D 109 – 23 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1923.

Properties.

1. The tar shall conform to the following requirements:[2]

(*a*) Water...0.00 per cent

(*b*) Float test at 32° C. (89.6° F.)..................60 to 150 sec.

(*c*) Distillation test:

Total Distillate, by weight, 0 to 170° C. (32 to 338° F.)	not more than	1.00	per cent
Total Distillate, by weight, 0 to 235° C. (32 to 455° F.)	not more than	4.00	"
Total Distillate, by weight, 0 to 270° C. (32 to 518° F.)	not more than	13.00	"
Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)	not more than	26.00	"
Residue, by weight	not less than	74.00	"

(*d*) Softening point (Ring-and-Ball Method) of residue from distillation test.......................not more than 65° C. (149° F.)

(*e*) Total Bitumen (Soluble in Carbon Disulfide)...not less than 95 per cent

Methods of Testing.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[3]

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] 1924 Book of A.S.T.M. Standards.

(*b*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20);[1]

(*c*) *Softening Point:* Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36);[1]

(*d*) *Float Test:* Tentative Method of Float Test for Bituminous Materials (Serial Designation: D 139 – 25 T);[2]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 – 23 T).[3]

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[1] 1924 Book of A.S.T.M. Standards.

[2] See p. 503.

[3] See p. 496.

TENTATIVE SPECIFICATIONS

FOR

HIGH-CARBON TAR CEMENT.[1]

Serial Designation: D 110 – 25 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1923, 1925.

Scope. 1. These specifications cover material suitable for use in the construction of tar macadam and tar concrete pavements.

Properties. 2. The tar cement shall conform to the following requirements:[2]

(*a*) Water ....0.00 per cent

(*b*) Softening point[3] (Ring-and-Ball Method) ....30 to 40° C. (86 to 104° F.)[4]

(*c*) Distillation test:

Total Distillate, by weight, 0 to 170° C. (32 to 338° F.)	not more than 1.00	per cent
Total Distillate, by weight, 0 to 270° C. (32 to 518° F.)	not more than 10.00	"
Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)	not more than 20.00	"
Residue, by weight	not less than 80.00	"

(*d*) Specific gravity at 25°/25° C. (77°/77° F.) of total distillate to 300° C. (572° F.) ....not less than 1.03

(*e*) Softening point (Ring-and-Ball Method) of residue from distillation test.......................not more than 65° C. (149° F.)

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] If desired, a float test may be substituted for the softening point test, in which case the requirements shall be as follows: Float Test at 50° C. (122° F.), 100 to 220 sec.

[4] The specification range for softening point, within the above limits, should be 5° C. for any given locality, for example, 30 to 35° C. for cold climates, equivalent to a float test at 50° C. of 100 to 160 sec.; 35 to 40° C. for warm climates, equivalent to a float test at 50° C. of 160 to 220 sec.

(*f*) Total Bitumen (Soluble in Carbon Disulfide)[1]..............
......................................78 to 95 per cent

Methods of Testing.

3. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[2]

(*b*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20;[2]

(*c*) *Softening Point:* Standard Method of Tests for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36);[2]

(*d*) *Float Test:* Tentative Method of Float Test for Bituminous Materials (Serial Designation: D 139 – 25 T);[3]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 – 23 T).[4]

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[1] The specification range for total bitumen covers a wide variety of materials. If products from vertical retort or low-carbon coke-oven tars are desired, a range of 88 to 95 per cent should be specified. If high-carbon coke-oven or mixtures of coke-oven and gas-house tars are desired, a range of 78 to 88 per cent should be specified.

[2] 1924 Book of A.S.T.M. Standards.

[3] See p. 503.

[4] See p. 496

TENTATIVE SPECIFICATIONS

FOR

LOW-CARBON TAR CEMENT[1]

Serial Designation: D 111 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1923.

Scope. 1. These specifications cover material suitable for use in the construction of tar macadam and tar concrete pavements.

Properties. 2. The tar cement shall conform to the following requirements:[2]

(*a*) Water....................................0.00 per cent

(*b*) Softening point[3] (Ring-and-Ball Method).................. ....30 to 40° C. (86 to 104° F.)[4]

(*c*) Distillation test:

Total Distillate, by weight, 0 to 170° C. (32 to 338° F.)..................	not more than 1.00	per cent
Total Distillate, by weight, 0 to 235° C. (32 to 455° F.)..................	not more than 2.00	"
Total Distillate, by weight, 0 to 270° C. (32 to 518° F.)..................	not more than 10.00	"
Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)..................	not more than 20.00	"
Residue, by weight..................	not less than 80.00	"

(*d*) Softening point (Ring-and-Ball Method) of residue from distillation test.......................not more than 65° C. (149° F.)

(*e*) Total Bitumen (Soluble in Carbon Disulfide)............. ....not less than 95 per cent

Methods of Testing. 3. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] If desired, a float test may be substituted for the softening point test, in which case the requirements shall be as follows: Float Test at 50° C. (122° F.), 100 to 220 sec.

[4] The specification range for softening point, within the above limits, should be 5° C. for any given locality, for example, 30 to 35° C. for cold climates, equivalent to a float test at 50° C. of 100 to 160 sec.; 35 to 40° C. for warm climates, equivalent to a float test at 50° C. of 160 to 220 sec.

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[1]

(*b*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20);[1]

(*c*) *Softening Point:* Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36);[1]

(*d*) *Float Test:* Tentative Method of Float Test for Bituminous Materials (Serial Designation: D 139 – 25 T);[2]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 – 23 T).[3]

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[1] 1924 Book of A.S.T.M. Standards.

[2] See p. 503.

[3] See p. 496.

TENTATIVE SPECIFICATIONS

FOR

COAL-TAR PITCH FOR STONE BLOCK FILLER[1]

Serial Designation: D 112 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1922, 1923.

Properties. 1. The coal-tar pitch shall conform to the following requirements:[2]

(*a*) Water....................................0.00 per cent

(*b*) Softening point[3] (Cube-in-Water Method)................. ....46 to 57° Cl (115 to 135° F.)

(*c*) Distillation test:

Total Distillate, by weight, 0 to 300° C. (32 to 572° F.)...................not more than 10.00 per cent.
Residue, by weight.................. not less than 90.00 "

(*d*) Specific gravity at 25°/25° C. (77°/77° F.) of total distillate to 300° C. (572° F.)...........................not less than 1.03

(*e*) Softening point (Cube-in-Water Method) of residue from distillation test....................not more than 75° C. (167° F.)

(*f*) Ductility at 50 to 100 penetration, at 25° C. (77° F.)....... ....not less than 50 cm.

Note.—The penetration of the pitch shall be brought within the range of 50 to 100 penetration by heating in an open vessel with frequent stirrings at a temperature of not over 350° F.

(*g*) Total Bitumen (Soluble in Carbon Disulfide)............... ....65.00 to 80.00 per cent.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] All tests shall be made on the sample as received, including the distillation test, and the results reported on a dry basis.

[3] The Softening Point (Cube-in-Water Method) specified should have a range of not over 10° F. within the above limits. The range, within the limits of 115 to 135° F. should vary with the use of the material, for example, if used in admixture with sand, in a northern locality or a southern locality. The softening point range, within the above limits, should also vary according to the character of the paving.

2. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials: **Methods of Testing.**

(*a*) *Percentage of Water:* Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95);[1]

(*b*) *Distillation:* Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20);[1]

(*c*) *Softening Point:* Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (Serial Designation: D 61);[1]

(*d*) *Ductility:* Tentative Method of Test for Ductility of Bituminous Materials (Serial Designation: D 113 – 22 T);[2]

(*e*) *Total Bitumen:* Tentative Method of Test for the Determination of Bitumen (Serial Designation: D 4 – 23 T).[3]

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[1] 1924 Book of A.S.T.M. Standards.

[2] See p. 501.

[3] See p. 496.

TENTATIVE METHOD OF TEST

FOR

SPECIFIC GRAVITY OF ROAD OILS, ROAD TARS, ASPHALT CEMENTS AND SOFT TAR PITCHES[1]

Serial Designation: D 70 – 20 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1920.

Definition. 1. The specific gravity of road oils, road tars, asphalt cements and soft tar pitches shall be expressed as the ratio of the weight of a given volume of the material at 25° C. (77° F.) to that of an equal volume of water at the same temperature and shall be expressed thus:

Specific gravity 25°/25° C. (77°/77° F.).

Apparatus. 2. The determination of specific gravity shall be made with a pyknometer or weighing bottle (Fig. 1), which shall consist of a straight-walled glass tube approximately 70 mm. long and 22 mm. in diameter, carefully ground to receive an accurately fitting solid glass stopper with a hole of 1.5 to 1.7-mm. bore in place of the usual capillary opening. The lower part of the stopper is made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shape depression shall be about 4.8 mm. at the center. The stoppered tube should have a capacity of about 24 cc. and when empty should weigh not over 35 g.

Procedure. 3. Before making a determination, the pyknometer with stopper shall first be calibrated by weighing it clean and dry upon an analytical balance. This weight is called "*a.*" It shall then be filled with freshly boiled distilled water at a temperature of 25° C. (77° F.), the stopper firmly inserted, all

[1] Criticisms of this Tentative Method are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

surplus moisture wiped from the surface with a clean dry cloth and again weighed. This weight is called "*b*."

4. When determining the specific gravity of road oils or road tars which flow readily, the material shall be brought to a temperature of 25° C. (77° F.) and poured into the pyknometer until it is full, with care to prevent the inclusion of air bubbles. The stopper is then firmly inserted and all excess of material forced through the opening is carefully removed with a clean dry cloth. The pyknometer and contents are then weighed and this weight is called "*c*." The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{c-a}{b-a}$$

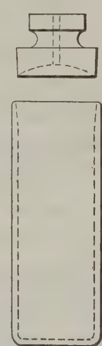

FIG. 1.—Pyknometer or Weighing Bottle.

5. When determining the specific gravity of tar and asphalt products which are too viscous for the method described in Section 4, a small amount of the material shall be brought to a fluid condition by the gentle application of heat, care being exercised to prevent loss by evaporation. When sufficiently fluid, enough is poured into the clean dry pyknometer to about half fill it. Precautions shall be taken to keep the material from touching the sides of the tube above the final level and to prevent the inclusion of air bubbles. The tube should be slightly warmed before filling. The pyknometer and contents are then cooled to room temperature and weighed with the stopper. This weight is called "*c*." The pyknometer is next removed from the balance, filled with freshly boiled distilled water, and the stopper firmly inserted. It is then completely immersed for not less than 30 minutes in a beaker of distilled water maintained at 25° C. (77° F.) after which it is removed, and all surplus water is wiped off with a clean cloth. It is immediately weighed. This weight is called "*d*." The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{c-a}{(b-a)-(d-c)}$$

6. When making the specific gravity determination it is important that: **Precautions.**

(*a*) Only freshly boiled distilled water shall be used.

(*b*) When weighing the pyknometer completely filled, the temperature of its contents shall be within 1° C. (1.8° F.) of 25° C. (77° F.).

(*c*) Precautions shall be taken to prevent expansion and overflow of the contents from the heat of the hand when wiping the surface of the pyknometer.

(*d*) The presence of all air bubbles shall be eliminated in filling the pyknometer and inserting the stopper.

(*e*) Weighings shall be made quickly after filling the pyknometer and shall be accurate to 1 mg. A number of trial fillings and catch weights may be necessary to obtain the desired degree of accuracy.

(*f*) To prevent breakage of the pyknometer when cleaning it out after a determination has been made upon a very viscous or semi-solid material, it will be found advisable to warm it in an oven at not over 100° C. until most of the material may be poured out and then to swab it with a piece of soft cloth or cotton waste. When cool it may be finally rinsed with carbon disulfide, benzol or other solvent and wiped clean.

Accuracy.

7. The limit of accuracy of the test is ±0.005 specific gravity.

TENTATIVE METHOD OF TEST
FOR
SPECIFIC GRAVITY OF ASPHALTS AND TAR PITCHES SUFFICIENTLY SOLID TO BE HANDLED IN FRAGMENTS.[1]

Serial Designation: D 71 – 20 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1920.

Definition. 1. The specific gravity of asphalts and tar pitches shall be expressed as the ratio of the weight of a given volume of the material at 25° C. (77° F.) to that of an equal volume of water at the same temperature and shall be expressed thus:

Specific Gravity 25°/25° C. (77°/77° F.).

Apparatus. 2. The determination of specific gravity shall be made with an analytical balance equipped with a pan straddle or other stationary support (Fig. 1).

Test Specimen. 3. The test specimen shall be a cube of the material measuring approximately $\frac{1}{2}$ in. to the edge. It shall be prepared by melting a small sample of the material by the gentle application of heat, care being exercised to prevent loss by evaporation, and pouring when sufficiently fluid into a $\frac{1}{2}$-in. brass cubical mold, which has been amalgamated with mercury and which is placed on an amalgamated brass plate. Precautions should be taken to prevent the inclusion of air bubbles. The hot material should slightly more than fill the mold and when cool the excess may be cut off with a hot spatula. The specimen shall be removed from the mold when cooled to room temperature.

Procedure. 4. The balance shall first be tared with a piece of fine waxed silk thread sufficiently long to reach from the hook on one of the pan supports to the straddle or rest. The test specimen

[1] Criticisms of this Tentative Method are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

shall then be attached to the thread, so as to be suspended about 1 in. above the straddle from the hook on the pan support, and weighed. This weight is called "*a*" and shall be accurate to 0.1 mg. The specimen, still suspended by the thread, shall then be weighed completely immersed in freshly boiled distilled water at 25° C. (77° F.) ±1° C. (1.8° F.), adhering air bubbles

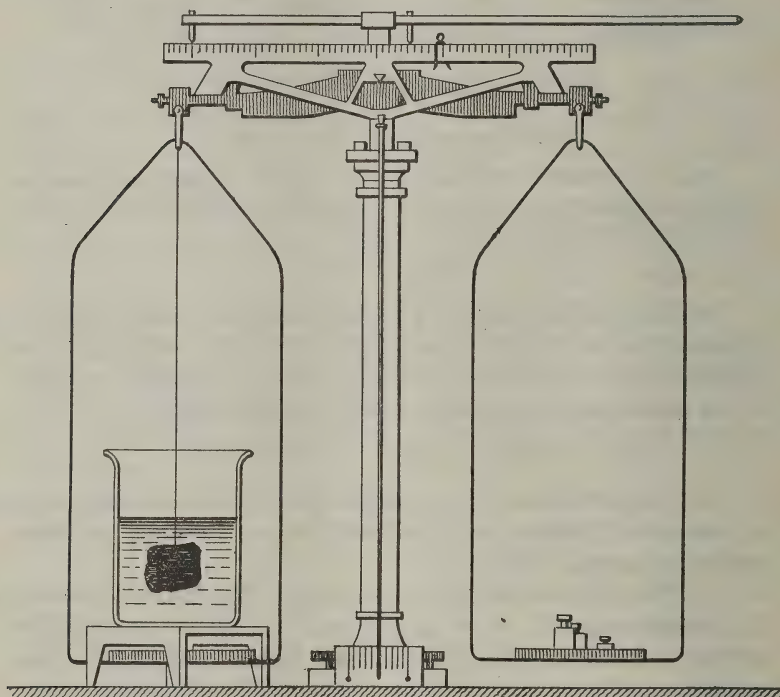

FIG. 1.—Analytical Balance Equipped with Pan Straddle.

being first removed with a fine wire. This weight is called "*b*" and shall also be accurate to 0.1 mg.

The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{a}{a-b}$$

Accuracy. 5. The limit of accuracy of the test is ±0.005 specific gravity.

TENTATIVE METHOD OF TEST

FOR

QUANTITY OF CLAY IN SAND-CLAY, TOPSOIL AND SEMI-GRAVEL FOR HIGHWAY CONSTRUCTION.[1]

Serial Designation: D 73 – 20 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1920.

Scope. 1. This test covers the determination of the quantity of clay in that portion of sand-clay, topsoil and semi-gravel for highway construction which passes a 10-mesh sieve.[2]

Treatment of Material. 2. Five hundred grams of material shall be dried to constant weight at a temperature below 176°.6 C. (350° F.). The material shall be gently pulverized to break down soft clods or masses, but not to grind or break hard material. It shall be passed through a 10-mesh sieve and the coarse residue weighed and recorded as "coarse material." The material passing the 10-mesh sieve shall be used as a starting point of a percentage analysis as follows:

Procedure. 3. Two samples of 50 g. of this material shall be weighed out for duplicate analysis. Each shall be placed in a tared wide-mouth bottle (5 to 6 cm. in diameter and 12 to 15 cm. high). Five cubic centimeters of dilute ammonia water and about 200 cc. of water shall be added. The bottle shall be closed with a cork or glass stopper and shaken vigorously for 20 minutes. The sample shall be allowed to settle 8 minutes and decanted carefully, or the supernatant liquid siphoned off to a depth of 8 cm. below the surface of the liquid. (The depth of the liquid in the bottle should be sufficient to leave about 4 cm. below the point of siphoning.) The bottle shall again be filled with water,

[1] Criticisms of this Tentative Method are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] For specifications for this sieve, see the Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, except Fine Aggregates Used in Cement Concrete Serial Designation: D 7), 1924 Book of A.S.T.M. Standards.

shaken for 3 minutes, allowed to settle and siphoned off as before. This process shall be repeated until the supernatent liquid is clear. Care should be taken to wash the stopper and neck of the bottle free from coarse material before decanting. The bottle and washed material shall be dried to constant weight at a temperature between 100 and 110° C. (212 to 230° F.), weighed, and the net weight of washed material determined.

Percentage of Clay. 4. The percentage of clay shall be calculated from the formula:

$$\text{Percentage of Clay} = \frac{\text{Original weight} - \text{weight after washing}}{\text{Original weight}} \times 100.$$

Check Determination. 5. As a check the washings drawn off shall be collected and evaporated to dryness for direct recovery of the fine sediment classed as clay:

$$\text{Percentage of Clay} = \frac{\text{Weight of residue}}{\text{Original weight}} \times 100.$$

Accuracy. 6. The determinations of percentage of clay on the two samples shall check within ± 1 percentage of clay to be acceptable.

TENTATIVE METHOD OF DECANTATION TEST
FOR
SAND AND OTHER FINE AGGREGATES.[1]

Serial Designation: D 136 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922.

Scope. 1. This method of test covers the determination of the total quantity of silt, loam, clay, etc., in sand and other fine aggregates.[2]

Apparatus. 2. The pan or vessel to be used in the determination shall be approximately 9 in. (230 mm.) in diameter and not less than 4 in. (102 mm.) in depth.

Treatment of Sample. 3. The sample must contain sufficient moisture to prevent segregation and shall be thoroughly mixed. A representative portion of the sample sufficient to yield approximately 500 g. of dried material, shall then be dried to a constant weight at a temperature not exceeding 110° C. (230° F.).

Procedure. 4. The dried material shall be placed in the pan and sufficient water added to cover the sample (about 225 cc.). The contents of the pan shall be agitated vigorously for 15 seconds, and then be allowed to settle for 15 seconds, after which the water shall be poured off, care being taken not to pour off any sand. This operation shall be repeated until the wash water is clear. As a precaution, the wash water shall be poured through a 200-mesh sieve and any material retained thereon returned to the washed sample. The washed sand shall be dried to a constant weight at a temperature not exceeding 110° C. (230° F.) and weighed.

Calculation of Results. 5. The results shall be calculated from the formula:

$$\text{Percentage of silt, clay, loam, etc.} = \frac{\text{Original dry weight} - \text{weight after washing}}{\text{Original dry weight.}} \times 100$$

Check Determination. 6. When check determinations are desired, the wash water shall be evaporated to dryness, the residue weighed, and the percentage calculated from the formula:

$$\text{Percentage of silt, loam, clay, etc.} = \frac{\text{Weight of residue}}{\text{Original dry weight}} \times 100$$

[1] Criticisms of this Tentative Method are solicited, and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] This determination of the percentage of silt, clay, loam, etc., will include all water-soluble material present, the percentage of which may be determined separately if desired.

TENTATIVE METHOD OF MECHANICAL ANALYSIS OF SUBGRADE SOILS.[1]

Serial Designation: D 137 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1922; Revised, 1923, 1924, 1925.

Scope.

1. This method covers the determination of the amount of sand, silt, clay and suspension clay[2] in subgrade soils.

Treatment of Sample.

2. The sample as received shall be dried in an oven at a temperature not to exceed 100° C. (212° F.) and shall then be broken up in a mortar by means of a rubber-covered pestle, care being taken not to break any fragments of rock or sand. The sample shall then be passed through a $\frac{1}{4}$-in. screen and the material passing this screen further pulverized by rolling out in a thin layer on a rubber pad, using a rubber-covered roller, after which the sample shall be passed through a 10-mesh sieve.

Apparatus.

3. The apparatus shall consist of a wide-mouthed cylindrical glass jar of at least 10 liters capacity; a soil centrifuge with a four or eight-tube head of such diameter that a speed of 1500 r.p.m. will exert a centrifugal force approximately 500 times the force of gravity; four centrifuge tubes of 100-cc. capacity; and a chemical balance sensitive to 0.001 g.

Procedure when Determination of Suspension Clay is Required.

4. By the method of quartering, a sample weighing approximately 25 g. and passing the 10-mesh sieve shall be selected and placed in a beaker with approximately 500 cc. of distilled water. This mixture shall be gradually brought up to the boiling point during a period of one hour and then be allowed to simmer for an additional hour. After standing until cool, the material in the beaker shall be thoroughly brushed and dispersed with a stiff brush for two or three minutes and then allowed to stand for eight minutes. The super-

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[1] Criticisms of this Tentative Method are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] The determination of the percentage of suspension clay by this method will include any water-soluble material present which may be determined separately if desired.

natant liquid shall then be decanted to a depth of 8 cm. from the surface of the liquid into a vessel having a capacity of about 10 liters. About 500 cc. of ammoniated water (concentration 1 : 500) shall be added to the material remaining in the beaker and the brushing repeated as before. After again standing for eight minutes, the supernatant liquid shall be decanted into the large vessel. This process shall be repeated until the supernatant liquid becomes clear after eight minutes sedimentation. The sand and silt have now been separated from the clay and suspension clay, which are contained in the large vessel. The material in the beaker shall then be transferred to an evaporating dish, dried to constant weight at a temperature not to exceed 100° C. (212° F.), cooled, and a mechanical analysis made, using 20-mesh, 60-mesh, 100-mesh and 200-mesh sieves. Material retained on each sieve shall be recorded as percentage of the original sample, and that which passes the 200-mesh sieve shall be recorded as the percentage of silt in the original sample.

The volume of liquid in the large vessel shall now be brought up to at least 10 liters by the addition of ammoniated water, and the vessel thoroughly shaken until all material is in suspension. Seventy cubic centimeters of the liquid shall then be siphoned from this vessel at a depth of about one-half the total depth of the liquid into each of four centrifuge tubes. The tubes shall then be mounted in the centrifuge and run for 30 minutes at a speed which, for the diameter of the head used, will exert a centrifugal force approximately 500 times the force of gravity. The material which has been thrown out of the liquid by centrifuging shall be classified as clay and that which remains in suspension as suspension clay. The solution containing the suspension clay shall then be decanted, evaporated to dryness, and the weight of the suspension clay determined. The clay remaining in the tube shall also be dried and weighed.

The weight of suspension clay is found from the liquid in four tubes, or 280 cc.; consequently this weight must be multiplied by the factor obtained by dividing the total volume used by 280 cc. in order to obtain the total suspension clay in the original sample. The weight of clay is determined from the clay in one tube, or 70 cc.; therefore the total volume divided by 70 cc. gives the factor by which this weight should be multiplied to give the total clay in the original sample. The total weights of clay and suspension clay thus obtained are converted into percentages of the original 25-g. sample. The determination of the percentage of suspension clay by this method includes any water-soluble material present which may be determined separately if desired.

Procedure when Determination of Suspension Clay is not Required.

5. In case it is not considered necessary to make a determination of the percentage of suspension clay, the following procedure for determination of total clay (clay plus suspension clay) may be followed:

An aliquot portion of the total volume of water used in washing the sample shall be taken and evaporated to dryness and the weight of clay plus suspension clay obtained, which shall be expressed as the percentage of total clay in the original 25-g. sample.

TENTATIVE METHOD OF TEST FOR THE DETERMINATION OF MOISTURE EQUIVALENT OF SUBGRADE SOILS IN THE FIELD[1]

Serial Designation: D 220 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope.

1. This method covers the determination of the moisture equivalent of subgrade soils by means of a field test.

Apparatus.

2. The apparatus shall consist of a bowl, a spatula, and a suitable device for adding water dropwise.

Treatment of Sample.

3. A sample, weighing approximately 500 g., shall be taken from the field, air dried, and the lumps reduced in size to approximately $\frac{1}{8}$ in., all rock above this size being removed.

Procedure.

4. (*a*) The soil sample (prepared as described in Section 3) shall be placed in a bowl and water slowly added from a burette, the water and soil being mixed by means of a spatula until the soil reaches the consistency of putty and so the soil may be compacted with the spatula without any free water remaining on the surface. The soil shall then be struck off smooth and a drop of water allowed to fall on the surface. If this drop is immediately absorbed by the soil it indicates that the moisture equivalent has not been reached. A small increment of water shall then be added and thoroughly mixed with the soil. The surface shall again be struck off smooth and an additional drop allowed to fall thereon. This process is repeated until upon the addition of a drop of water the surface remains moist and has a shiny appearance.

By adding small increments of water and by watching the surface of the soil carefully, the critical point may readily be determined.

(*b*) The sample shall then be dried out at a temperature not exceeding 100° C. and the percentage of moisture determined on the basis of the dry weight of the sample.

NOTE.—This test is inaccurate when applied to soils whose moisture equivalent is below 20.

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

TENTATIVE METHOD OF TEST

FOR

CONSISTENCY OF PORTLAND-CEMENT CONCRETE[1]

Serial Designation: D 138 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED 1925.

Scope.

1. This test covers the method to be used both in the laboratory and in the field for determining consistency of concrete[2].

Apparatus. 2. The test specimen shall be formed in a mold of No. 16 gage galvanized metal in the form of the lateral surface of the frustrum of a cone with the base 8 in. in diameter, the upper surface 4 in. in diameter, and the altitude 12 in. The base and the top shall be open and parallel to each other and at right angles to the axis of the cone. The mold shall be provided with foot pieces and handles as shown in Fig. 1.

Sample. 3. When the test is made at the mixer, the sample shall be taken from the pile of concrete immediately after the entire batch has been discharged. When testing concrete that has been hauled from a central mixing plant, the sample shall be taken from the concrete immediately after it has been dumped on the subgrade.

Procedure. 4. The mold shall be placed on a flat, non-absorbent surface, such as a smooth plank or a slab of concrete, and the operator shall hold the form firmly in place, while it is being filled, by standing on the foot pieces. The mold shall be filled to about one-fourth of its height with the concrete which shall then be puddled, using 20 to 30 strokes of a $\frac{1}{2}$-in. rod pointed at the lower end. The filling shall be completed in successive layers similar to the first and the top struck off so that the mold is exactly filled. The mold shall then be removed by being

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[1] Criticisms of this Tentative Method are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] This test is not considered applicable when there is a considerable amount of coarse aggregate over 2 in. in size in the concrete. The committee is now working on a method suitable for determining the consistency of concrete using aggregate over 2 in. in size.

raised vertically, immediately after being filled. The molded concrete shall then be allowed to subside until quiescent and the height of the specimen measured.

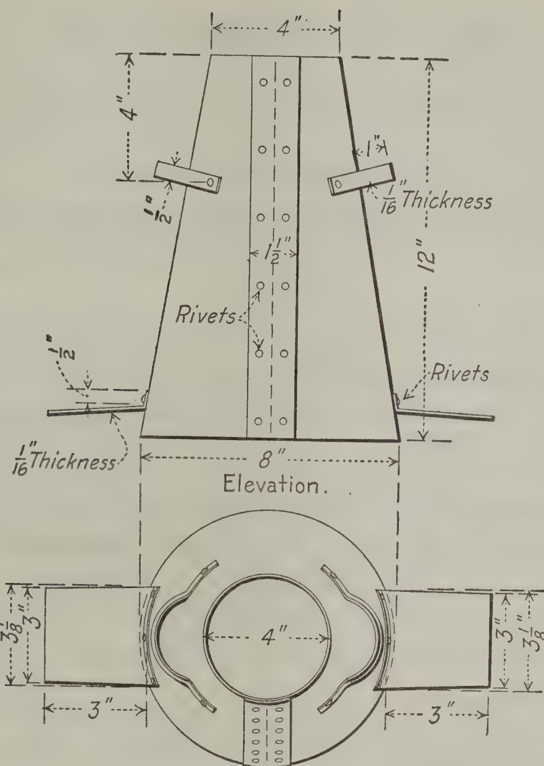

FIG. 1.

5. The consistency shall be recorded in terms of inches of subsidence of the specimen during the test, which shall be known as the slump. **Slump.**

Slump = 12 − inches of height after subsidence

TENTATIVE METHOD OF TEST

FOR

THE DETERMINATION OF BITUMEN[1]

Serial Designation: D 4 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923

Scope. 1. Bitumen may usually be expeditiously and accurately determined by Method No. 1, Section 5. In the case of certain native asphalts containing finely divided mineral matter, the mineral residue is not easily retained by the filter and it is necessary to resort to Method No. 2, Section 6, in order to obtain accurate results. Method No. 2 shall not be used unless the filter clogs unduly or unless the mineral matter passing through the filter exceeds 0.5 per cent. In every case the report shall indicate whether Method No. 1 or Method No. 2 has been employed.

I. APPARATUS

2. The apparatus shall consist of the following:

(*a*) Gooch crucible, approximately 4.4 cm. in width at the top, tapering to 3.6 cm. at the bottom, with a depth of 2.5 cm.;

(*b*) Filtering flask;

(*c*) Rubber stopper;

(*d*) Filter tube;

(*e*) Section of rubber tubing to hold the Gooch crucible on the filter tube;

(*f*) Asbestos (amphibole) cut in pieces not exceeding 1 cm. in length, shredded and shaken up with water;

(*g*) Two 150-cc. Erlenmeyer flasks;

(*h*) Bunsen burner;

(*i*) Drying oven;

(*j*) Suction pump;

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

This method is in effect a revision of the Standard Method of Test for Soluble Bitumen. The Standard Method, which was last published under the Serial Designation: D 4 – 11, has accordingly been withdrawn.

(*k*) Rubber tubing;
(*l*) Analytical balance;
(*m*) Desiccator.

II. PREPARATION OF SAMPLE

3. The sample shall be representative and if it contains more than 2 per cent of water it shall be dehydrated by distillation in a copper still in accordance with the Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20) of the American Society for Testing Materials,[1] the water-free distillate being returned to the residue. If the material is hard and brittle, it may be ground and dried at a temperature below the temperature of volatilization of the material.

III. PROCEDURE

Preparation of Gooch Crucible.

4. The Gooch crucible shall be set in the filter tube inserted in the stopper of the filtering flask. The flask shall be connected with the suction pump. Before suction shall be applied, the crucible shall be filled with asbestos suspended in water which shall be allowed to partly settle in the crucible. A light suction shall be applied to draw off the water, leaving a firm mat of asbestos in the crucible. More suspended asbestos shall be added and the process repeated until a felt is built up that barely transmits light. The felt shall then be thoroughly washed with water, dried in a drying oven, and ignited over a Bunsen burner. The crucible shall then be cooled in a desiccator and weighed.

(*a*) *Method No. 1*

Procedure No. 1.

5. An amount of material which shall contain approximately one gram of bitumen shall be weighed into a tared Erlenmeyer flask. One hundred cubic centimeters of chemically pure carbon disulfide shall be added to the flask in small portions with continued agitation until all lumps disappear and nothing adheres to the bottom. The flask shall be corked and set aside for fifteen minutes.

The Gooch crucible shall be set up again with the suction flask and the carbon disulfide solution carefully decanted through the asbestos felt. No sediment shall be allowed to go onto the filter. A small amount of carbon disulfide shall be used to wash down the sides of the flask and then the precipitate shall be brought onto the felt and the flask scrubbed with a feather if necessary to remove all precipitate. The contents of the crucible shall be washed with carbon disulfide until the washings are colorless. Suction shall be applied to the

[1] 1924 Book of A.S.T.M. Standards.

crucible to remove the carbon disulfide. The crucible shall be dried in the oven at 100 to 125° C. for twenty minutes, cooled in the desiccator and weighed.

In case insoluble matter adheres to the flask, the flask shall be dried and weighed and the increase in weight over the original weight shall be added to the weight of insoluble matter in the crucible.

The crucible shall be ignited at a red heat and after thorough ignition, cooled and weighed.

The weight of substance taken, minus the total weight of matter insoluble in carbon disulfide, is the total bitumen. The weight of material after ignition is ash.

(*b*) *Method No. 2*

Procedure No. 2.

6. Material shall be weighed out in the same way as in (*a*) Method No. 1 (Section 5) into a tared Erlenmeyer flask, and treated with 100 cc. of chemically pure carbon disulfide.

The flask shall be loosely corked and shaken at intervals until all large particles of material have been broken down. The flask shall be left undisturbed for 48 hours. The solution shall be decanted into a similar tared flask, care being taken to disturb as little of the residue as possible. The first flask shall be treated again with fresh carbon disulfide as before and left undisturbed for 48 hours. The solution from the second flask shall then be carefully decanted upon the Gooch crucible without use of vacuum, and this shall be followed by the solution from the first flask. The filter shall be washed with fresh carbon disulfide. The residue remaining in each flask shall be shaken again with fresh carbon disulfide and allowed to settle for 24 hours. The solution from both flasks shall then be decanted through the filter and the residues remaining in the flasks shall be washed again with carbon disulfide, the solution decanted and the process repeated until the washings are practically colorless. The temperature shall be maintained between 20 and 25° C.

The crucible and both flasks shall be dried at 100 to 125° C. and weighed. The filtrate containing the bitumen shall be evaporated, the bituminous residue burned, and the weight of the ash thus obtained added to that of the residue in the two flasks and the crucible. The sum of these weights deducted from the weight of substance taken is the weight of bitumen.

TENTATIVE METHOD OF TEST

FOR

THE DETERMINATION OF PROPORTION OF BITUMEN SOLUBLE IN CARBON TETRACHLORIDE[1]

Serial Designation: D 165 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

Issued, 1923

I. APPARATUS

1. The apparatus shall consist of the following: **Apparatus.**

(*a*) Gooch Crucible, approximately 4.4 cm. in width at the top, tapering to 3.6 cm. at the bottom, with a depth of 2.5 cm.

(*b*) Filtering flask;

(*c*) Rubber stopper;

(*d*) Filter tube;

(*e*) Section of rubber tubing to hold the Gooch crucible on the filter tube;

(*f*) Asbestos (amphibole) cut in pieces not exceeding 1 cm. in length, shredded and shaken up with water;

(*g*) Two 150-cc. Erlenmeyer flasks;

(*h*) Bunsen burner;

(*i*) Drying oven;

(*j*) Suction pump;

(*k*) Rubber tubing;

(*l*) Analytical balance;

(*m*) Desiccator.

II. PREPARATION OF SAMPLE

2. The sample shall be representative and if it contains more than 2 per cent of water it shall be dehydrated by distillation in a copper still in accordance with the Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (Serial Designation: D 20) of the American Society for Testing Materials,[2] and water-

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] 1924 Book of A.S.T.M. Standards.

free distillate returned to the residue. If the material is hard and brittle, it may be ground and dried at a temperature below the temperature of volatilization of the material.

III. PROCEDURE

Preparation of Gooch Crucible.

3. The Gooch crucible shall be set in the filter tube inserted in the stopper of the filtering flask. The flask shall be connected with the suction pump. Before suction is applied, the crucible shall be filled with asbestos suspended in water which shall be allowed to partly settle in the crucible. A light suction shall be applied to draw off the water, leaving a firm mat of asbestos in the crucible. More suspended asbestos shall be added and the process repeated until a felt is built up that barely transmits light. The felt shall then be thoroughly washed with water, dried in a drying oven, and ignited over a Bunsen burner. The crucible then shall be cooled in a desiccator and weighed.

Procedure.

4. An amount of material which shall contain approximately one gram of bitumen shall be weighed into a tared Erlenmeyer flask. One hundred cubic centimeters of chemically pure carbon tetrachloride shall be added to the flask in small portions with continued agitation until all lumps disappear and nothing adheres to the bottom. The flask shall be corked and set aside in subdued light for at least 12 hours.

The Gooch crucible shall be set up again with the suction flask and the carbon tetrachloride solution carefully decanted through the asbestos felt. No sediment shall be allowed to go onto the filter. A small amount of carbon tetrachloride shall be used to wash down the sides of the flask and then the precipitate shall be brought onto the felt and the flask scrubbed with a feather if necessary to remove all precipitate. The contents of the crucible shall be washed with carbon tetrachloride until the washings are colorless. Suction shall be applied to the crucible to remove the carbon tetrachloride. The crucible shall be dried in the oven at 100 to 125 C. for twenty minutes, cooled in the desiccator, and weighed.

In case insoluble matter adheres to the flask, the flask shall be dried and weighed and the increase in weight over the original weight shall be added to the weight of insoluble matter in the crucible.

The proportion of bitumen soluble in carbon tetrachloride shall be reported on the basis of total bitumen taken as 100.

$$\text{Proportion of Bitumen Soluble in Carbon Tetrachloride} = \frac{\text{Bitumen Soluble in Carbon Tetrachloride}}{\text{Total Bitumen}}$$

TENTATIVE METHOD OF TEST
FOR
DUCTILITY OF BITUMINOUS MATERIALS.[1]

Serial Designation: D 113 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1922.

Definition. 1. The ductility of an asphalt cement or semi-solid bitumen is the distance to which it will elongate before breaking when two ends of a briquette of the material are pulled apart at a specified rate of speed and at a specified temperature. When the conditions of the test are not specifically mentioned, the rate and the temperature are understood to be 5 cm. per minute at 25° C. (77° F.).

APPARATUS.

Mold. 2. The mold shall be as shown in Fig. 1. It shall be made of brass, the ends, b and b′, being known as clips, and the parts, a and a′, as sides of the mold. The dimensions of the mold shall be as follows:

Total length (internal)	7.45 – 7.55 cm.
Distance between clips	2.97 – 3.06 "
Width of clips at mouth	1.98 – 2.02 "
Width of briquette at minimum cross-section (half way between clips)	0.99 – 1.01 "
Thickness of briquette throughout	0.99 – 1.01 "

Water Bath. 3. The water bath shall be maintained at a temperature not varying more than 0.1° C. from 25° C. (77° F.). The volume of water shall be not less than 10 liters and the sample shall be immersed to a depth of not less than 10 cm. and shall be supported on a perforated shelf not less than 5 cm. from the bottom of the bath.

Testing Machine. 4. Any apparatus may be used for pulling the briquette of bitumen apart that is so constructed that the briquette will be continuously immersed in water and the two clips pulled apart at a uniform rate of speed of 5 cm. per minute.

[1] Criticisms of this Tentative Method are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

PROCEDURE.

Melting and Molding.

5. The asphalt cement or bituminous material to be tested shall be completely melted at such a temperature that it will be thoroughly fluid—with ordinary paving asphalt cement this is about 160° C. (320° F.). It shall then be strained through a 50-mesh sieve and, after a thorough stirring, poured into the mold. The mold shall be assembled on a brass plate and so as to prevent the material under test from sticking, the surface of the plate and the interior surfaces of the side pieces *aa′* of the mold shall be thoroughly amalgamated. In filling the mold care shall be taken not to disarrange the parts and thus distort the briquette. In filling, the material shall be poured in a thin stream back and forth from end to end of the mold until it is more than level full. It shall be left to cool to room temperature and then placed in water maintained at 25° C. (77° F.) for 30 minutes, after which the excess of bitumen shall be cut off by means of a hot putty knife or spatula so that the mold shall be just level full.

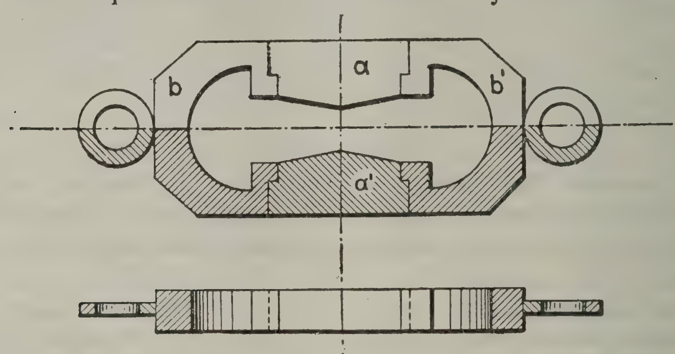

FIG. 1.—Ductility Mold.

Keeping at Standard Temperature.

6. The brass plate and mold, with briquette, shall then be placed in the water bath and kept at a temperature of 25° C. (77° F.) for at least 1½ hours, when the briquette shall be removed from the plate and the side pieces detached.

Testing.

7. The rings at each end of the clip shall then be attached to the pins or hooks in the ductility machine and the two clips pulled apart at a uniform rate of 5 cm. per minute (±5 per cent). While the test is being made, the water in the tank of the ductility machine shall cover the sample by at least 2.5 cm. and shall be kept continuously at a temperature within 0.5° C. of 25° C. (77° F.). When the specimen of bituminous material breaks, the distance from the original position of the clip before pulling to its present position shall be measured, and shall be known as the ductility. The average of three tests shall be taken, excepting that any abnormal result shall be rejected.

TENTATIVE METHOD OF FLOAT TEST FOR BITUMINOUS MATERIALS[1]

Serial Designation: D 139 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923, 1924, 1925.

I. APPARATUS.

Float. 1. The float (Fig. 1) shall be made of aluminum or aluminum alloy and shall be in accordance with the following requirements:

	MINIMUM.	NORMAL.	MAXIMUM.
Weight of float, g	37.70	37.90	38.10
Total height of float, mm	34.0	35.0	36.0
Height of rim above lower side of shoulder, mm	26.5	27.0	27.5
Thickness of shoulder, mm	1.3	1.4	1.5
Diameter of opening, mm	11.0	11.1	11.2

Collar. 2. The collar (Fig. 1) shall be made of brass and shall be in accordance with the following requirements:

	MINIMUM.	NORMAL.	MAXIMUM.
Weight of collar, g	9.60	9.80	10.00
Over-all height of collar, mm	22.3	22.5	22.7
Inside diameter at bottom, mm	12.72	12.82	12.92
Inside diameter at top, mm	9.65	9.70	9.75

The top of the collar shall screw up tightly against the lower side of the shoulder.

Assembly. 3. The assembled float and collar, with the collar filled flush with the bottom and weighted to a total weight of 53.2 g., shall float upon water with the rim 8.5 ± 1.5 mm. above the surface of the water. Dimensions of the apparatus additional to those required above are given in Fig. 1.

Thermometer. 4. The thermometer shall conform to the following requirements. These specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 0 to 80° C. or 30 to 180° F., respectively.

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISION: −2 to +80° C. in 0.2° C. or +30 to +180° F. in 0.5° F.

[1] Criticisms of this Tentative Method are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

TOTAL LENGTH: 378 to 384 mm. (14.88 to 15.12 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.0 to 7.0 mm. (0.24 to 0.28 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 9 to 14 mm. (0.35 to 0.55 in.).
Diameter, 4.5 to 5.5 mm. (0.18 to 0.22 in.).

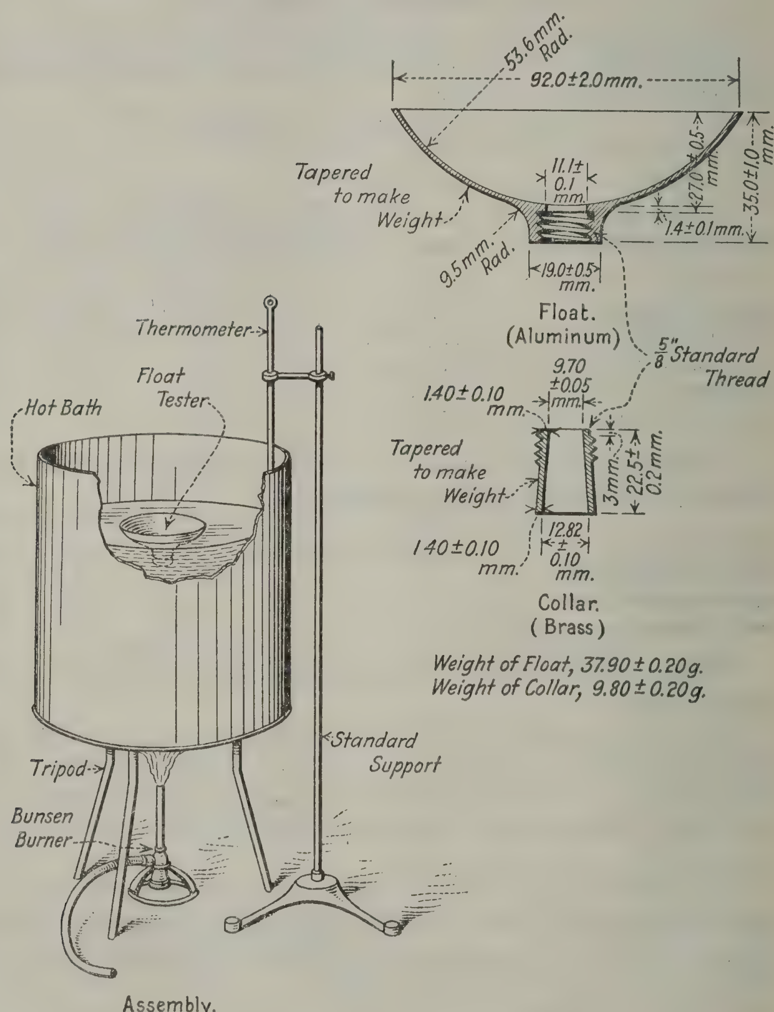

FIG. 1.—Float Test Apparatus.

DISTANCE TO 0° C. OR 32° F. LINE FROM BOTTOM OF BULB: 75 to 90 mm. (2.95 to 3.54 in.).

DISTANCE TO 80° C. OR 176° F. LINE FROM TOP OF THERMOMETER: 30 to 45 mm. (1.18 to 1.77 in.).

EXPANSION CHAMBER: To permit heating the thermometer at least 50° C. (90° F.) above highest temperature on scale.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass Ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. Each whole degree Centigrade or Fahrenheit line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2° C. or 5° F.

IMMERSION: Total.

SPECIAL MARKING: "A.S.T.M. Low S.P.," a serial number and the manufacturer's name or trade mark shall be etched on the thermometer.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.2° C. or 0.4° F.

STANDARDIZATION: The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, at the ice point and at temperature intervals of approximately 20° C. or 40° F.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Low S.P., 0 to 80° C." or "A.S.T.M. Low S.P., 30 to 180° F." according to the type of thermometer.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

Bath.

5. The diameter of the bath and the depth of water shall be at least 185 mm.

II. PREPARATION OF SAMPLE.

6. The brass collar shall be placed with the smaller end on a brass plate which has been previously amalgamated with mercury by first rubbing it with a dilute solution of mercuric chloride or nitrate, and then with mercury.

Preparation of Sample.

The sample shall be completely melted at the lowest possible temperature that will bring it to a sufficiently fluid condition for pouring, excepting creosote oil residues, which shall be mixed and poured at a temperature of 100 to 125° C. It shall be stirred thoroughly until it is homogeneous and free from air bubbles. The sample shall then be poured into the collar in any convenient manner until slightly more than level with the top.

Asphalt and Asphalt Products.—Asphalt and asphalt products shall be cooled to room temperature, placed in water maintained at 5° C. for 5 minutes, after which the surplus material shall be removed by means of a spatula, or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5° C., ±1° C., and left in this bath for at least 15 minutes.

Tar Products.

Tar Products.—Tar products shall be immediately immersed in ice water maintained at 5° C. for 5 minutes, after which the surplus material shall be removed by means of a spatula or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5° C., ±1° C., and left in this bath for at least 15 minutes.

III. PROCEDURE.

Procedure.

7. (*a*) The bath shall be filled with water and the water heated to the temperature at which the test is to be made. This temperature shall be accurately maintained and shall at no time throughout the test be allowed to vary more than 0.5° C. from the temperature specified.

(*b*) After the material to be tested has been kept in the ice water for not less than 15 minutes nor more than 30 minutes, the collar with its contents shall be removed from the plate and screwed into the aluminum float and immersed in water at 5° C. for one minute. Any water shall then be removed from the inside of the float and the latter immediately floated in the warm bath. As the plug of material becomes warm and fluid, it is forced upward and out of the collar until the water gains entrance into the saucer and causes it to sink.

(*c*) The time in seconds between placing the apparatus on the water and when the water breaks through the material shall be determined by means of a stop watch, and shall be taken as a measure of the consistency of the material under examination.

NOTE.—Special precaution should be taken to insure the collar fitting tightly into the float and to see that there is no seepage of water between the collar and float during the test.

TENTATIVE METHOD OF TEST

FOR

FINENESS OF POWDERED COAL[1]

Serial Designation: D 197 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society, and is subject to annual revision.

ISSUED, 1924

I. APPARATUS

Sieves. 1. Sieves Nos. 40, 50, 70, 100, 140, and 200 of the United States Standard Sieve Series,[2] with close-fitting pans and covers, shall be used.

Containers. 2. (*a*) *For Collecting Gross Sample.*—The containers shall be of metal of about 8 to 10-liter capacity, provided with tightly fitting covers.

NOTE.—Two-gallon milk cans are suitable for this purpose.

(*b*) *For Collecting Laboratory Sample.*—The containers may be of metal or glass of about one-liter capacity, provided with tightly fitting covers.

II. SAMPLING

Unit of Sampling. 3. The coal pulverized, per mill, during the period of one hour shall be considered a unit for sampling.

A. Multiple System

In the multiple powdered coal system, the coal after pulverization is conveyed into bins.

Sampling. 4. Not less than 2 kg. of representative powdered coal shall be collected as it is being discharged from the pulverizer. This is best accomplished by collecting increments of not more than 200 g. at regular intervals by means of a scoop. The increments shall be placed into a can of about 8 to 10-liter capacity, provided with a tightly fitting cover.

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. W. A. Selvig, Secretary of Committee D-5 on Coal and Coke, U. S. Bureau of Mines, 4800 Forbes St., Pittsburgh, Pa.

[2] For detailed specifications for these sieves, see U. S. Bureau of Standards *Letter Circular No. 74.*

Preparation of Sample. 5. Th,he grosssample shall be placed on a sheet of rubber or oil-cloth, and thoroughly mixed by raising first one corner of the cloth and then the other so as to roll the coal over and over at least 100 times. After mixing, the sample shall be coned and quartered. The operations of mixing, coning, and quartering shall be continued if necessary until the sample is reduced sufficiently so that all of one of the quarters weighs about 500 g., which coal shall constitute the laboratory sample. The laboratory sample shall be transferred to a metal or glass container of about one-liter capacity, provided with a tightly fitting cover.

NOTE.—The operations of mixing, coning and quartering are described and illustrated in the Standard Methods of Sampling Coal (Serial Designation: D 21) of the American Society for Testing Materials.[1]

B. Unit System

In the unit powdered coal system, the coal during and after being pulverized is at once mixed with air and delivered to the furnace. It is difficult to obtain representative samples, as it is necessary to sample the coal from a moving stream of coal and air inside the pipe between the pulverizer and the furnace. It is preferable to collect such samples from vertical pipes if possible, as in horizontal pipes considerable segregation takes place, the coarser particles tending to flow along the bottom of the pipes.

Sampling Device. 6. (*a*) The sampling device shall consist of a 1-in. thin-walled brass tube, with one end bent at a right angle or provided with a 90-deg. elbow and short length of tube. At the other end of the sampling tube a vacuum-sweeper bag shall be tightly attached. The inside of the sampling tube shall have no projections to interfere with the flow of coal.

(*b*) The sampling tube shall be inserted into the powdered coal pipe through a stuffing-box in such a manner that the opening of the sampling tube shall be directed against the stream of coal and air, and permit moving back and forth across the entire diameter of the coal pipe. If the sample must be taken from a horizontal pipe, the sampling tube shall be inserted through the top of the coal pipe, in such a manner as will permit moving the tube back and forth from top to bottom along the vertical diameter of the pipe.

Sampling. 7. In collecting the sample, the sampling tube shall be moved uniformly back and forth along the diameter of the pipe. In order to permit ready elimination of air from the bag, the vacuum-cleaner bag shall not be filled more than about one-fifth full at any time. Sampling shall be continued at regular intervals until a gross sample of not less than 5 kg. of coal is collected.

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[1] 1924 Book of A.S.T.M. Standards.

8. The gross sample shall be mixed, coned and quartered, and reduced in the same manner as described for the multiple powdered coal system. **Preparation of Sample.**

III. PROCEDURE

9. The entire laboratory sample shall be air dried in a drying oven at 10 to 15° C. above room temperature. The drying shall be continued until the loss in weight is not more than 0.1 per cent per hour. **Drying.**

10. After air drying, the sample shall be thoroughly mixed by rolling over and over at least 100 times on a sheet of rubber or oilcoth, as described for the mixing of the gross sample. The sample, after mixing, shall be spread out to a depth of about ½ in. and portions, representing a section from the top of the coal to the mixing cloth, shall be taken with a spatula at regular intervals. **Reducing Sample.**

11. A 50-g. sample of the air-dried coal shall be placed on the No. 200 sieve, to which a pan and cover shall be attached. The sieve shall be held in one hand in a slightly inclined position, so that the sample will be well distributed over the sieve, while at the same time the operator shall gently strike the side about 150 times per minute against the palm of the other hand on the up stroke. Every 25 strokes the sieve shall be turned about one-sixth of a revolution in the same direction. The sieve shall be held over a sheet of glazed white paper during the sieving operation, so as to detect any loss that might result from accidental spilling of a portion of the sample, in which case another sample shall be taken. **Procedure.**

After sieving for one minute, the sieve pan shall be removed, and the material which has passed through the sieve discarded. The side of the sieve shall be tapped with a brush handle, and the coal adhering to the under side of the wire cloth brushed off. During the operation of tapping and brushing, the sieve cover shall remain on the sieve so as to prevent any loss of material over the side of the sieve.

NOTE.—A 1-in. bristle brush is satisfactory for brushing the under side of the wire cloth.

The sieve pan shall be replaced, and the sieving continued for two minutes; then the tapping and brushing shall be repeated, and again repeated after two minutes of additional sieving. The operation shall then be continued in the usual manner, omitting the tapping and brushing, until the amount passing through in one minute of continuous sieving is about 0.1 g. Each time, before weighing the material passing through the sieve, the side of the sieve shall be tapped with the brush handle in order to remove any material adhering to

the under side of the wire cloth. The cover of the sieve shall then be removed, and the residue remaining on the sieve carefully removed. The sieve shall be inverted over a piece of glazed white paper, and the wire cloth cleaned by carefully brushing the under side. The material thus removed from the wire cloth shall be added to the residue removed from the sieve.

After cleaning the sieve, the residue shall be placed thereupon, the sieve pan and cover attached, and the sieving resumed in the usual manner, omitting the operations of tapping and brushing, until not more than 0.05 g. passes through in one minute of continuous sieving, when the sieving shall be considered to be finished. The sieve shall be inverted and cleaned as before, and the residue weighed.

12. The residue shall then be successively sieved on the Nos. 140, 100, 70, 50, and 40 sieves. Sieving shall be continued in the usual manner, omitting the tapping and brushing, until not more than 0.05 g. passes through in one minute of continuous sieving, when the sieving shall be considered finished. The sieve shall then be inverted, cleaned, and the residue weighed as described for the No. 200 sieve.

NOTE.—As the fine material which tends to clog the meshes of the sieves has been removed by sieving through the No. 200 sieve, it is not necessary to brush the under side of the wire cloth or clean the sieves during the sieving operation, as was done for the No. 200 sieve.

13. The fineness shall be calculated from the weights of the residues on the sieves, and shall be expressed as percentages of the weight of the original sample. Sieving value corrections for the No. 200 sieves, as determined by the U. S. Bureau of Standards, shall be applied to the material sieved on the No. 200 sieves. The fineness test shall be reported as follows:

	Per Cent
Retained on No. 40 sieve	----------
" " No. 50 sieve, passing No. 40 sieve	----------
" " No. 70 sieve, " No. 50 sieve	----------
" " No. 100 sieve, " No. 70 sieve	----------
" " No. 140 sieve, " No. 100 sieve	----------
" " No. 200 sieve, " No. 140 sieve	----------
" No. 200 sieve	----------

NOTE.—In applying the sieving value corrections, the graphical or analytical methods for obtaining such corrections for sieves which have been tested with a single standard sample shall be used. These methods are described in U. S. Bureau of Standards *Technologic Paper No. 42*, pp. 45–47 (1914).

Duplicate Determinations.

14. Duplicate determinations by the same operator, using the same sieves, shall check within 1 per cent on all sizes. Different

operators, using different sieves, shall check within 3 per cent on the material sieved on the No. 200 sieves.

NOTE.—Until such time as standard materials are available for determining sieving values of Nos. 140, 100, 70, 50, and 40 sieves, it is not feasible to specify check imits for the sizes as determined with these sieves, when different sieves are used.

RAPID ROUTINE METHOD BY MACHINE SIEVING

For routine testing, mechanical sieving devices, by which a number of sieves are shaken at the same time, afford a convenient method of making fineness tests. The hand-sieving method shall however be the standard method of test. The results obtained by machine sieving should be compared to those obtained by hand sieving, to determine whether the sieving action of the machine used is practically the same as by hand sieving, for the coal being sieved.

The same sieves should be used as specified for hand sieving, and these sieves should be provided with well-fitting pans and covers to prevent possible loss of material during sieving. A 50-g. sample of air-dried coal should be placed on each sieve, and the sieves shaken until not more than about 0.1 g. passes through the finest sieve of the series in the machine in one minute of continuous sieving. The sieves should then be inverted and brushed clean, as described for the No. 200 sieve in the method for hand sieving. Sieving should then be resumed until not more than 0.05 g. passes through the finest sieve of the series in the machine in one minute of continuous sieving, when the test shall be considered finished, after which the sieves should be inverted and cleaned as before, and the residues remaining on the sieves weighed. The fineness should be calculated and reported as in the hand-sieving method.

TENTATIVE DEFINITIONS

OF

TERMS RELATING TO COKE[1]

Serial Designation: D 121 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1921; REVISED, 1924

Coke.—A solid carbonaceous fuel obtained, as a coherent cellular residue, by the destructive distillation of coal and certain other carbonaceous substances such as coal tar and petroleum pitches.

NOTE.—In addition to carbon, as its principal constituent, it contains moisture, mineral matter (ash) and small amounts of residual volatile matter, nitrogen, and sulfur.

Beehive Coke.—Coke manufactured in beehive, rectangular or similar forms of ovens, in a horizontal bed covering the floor of such oven, where the temperature for distillation is usually secured by the combustion, within the chamber, of a portion of the material treated, the volatile matter being lost.

NOTE.—These ovens are heated by admission of a limited quantity of air, producing sufficient combustion to coke the coal in a specified time. In some instances heating flues are provided wherein a portion of the evolved gases are consumed. The underlying principle in the heating of the charge is internal combustion of the gases in the crown or dome of the oven, which plays an important part as a means for storage of heat and radiation to the coking mass.

By-Product Coke.—Coke manufactured in chambers or ovens, substantially rectangular in section, the height and length being materially greater than the width, and the chambers being heated externally, so that the volatile matter is saved in the form of by-products.

NOTE.—These ovens are heated by burning gas in the flues in the oven walls, the gas being either part of the coke-oven gas or derived from an external source. The underlying principle of the heating of the charge is external combustion, the heat so generated passing through the oven walls to the coking mass. Coke is the primary product, the volatile by-products being made available by suitable recovery apparatus.

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[1] Criticisms of these Tentative Definitions are solicited and should be directed to Mr. W. A. Selvig, Sercetary of Committee D-5 on Coal and Coke, U. S. Bureau of Mines, 4800 Forbes St., Pittsburgh, Pa.

Gas House Coke.—Coke obtained from retorts as a by-product in the manufacture of coal gas.

Beehive Foundry Coke.—Beehive coke which is physically and chemically suitable for use in foundry cupola practice.

NOTE.—Beehive foundry coke may be either 72-hour or 48-hour coke, the former differing from the latter mainly in the greater size of its pieces.

By-Product Foundry Coke.—By-product coke which is physically and chemically suitable for use in foundry cupola practice.

NOTE.—Such coke passes over a screening device having approximately $2\frac{1}{2}$-in. openings. It is not produced on any standard coking time.

Beehive Furnace Coke.—Beehive coke which is physically and chemically suitable for the reduction of iron ore in blast furnaces.

NOTE.—The breeze or fine coke smaller than approximately $\frac{3}{4}$ in. is forked or screened out. The usual coking time on a normal working schedule is four 48-hour charges and two 72-hour charges.

By-Product Furnace Coke.—By-product coke which is physically and chemically suitable for the reduction of iron ore in blast furnaces.

NOTE.—Such coke passes over a screening device with openings varying from $\frac{3}{4}$ to $1\frac{1}{2}$ in., depending on local conditions at different plants. It is not produced on any standard coking time.

Coke Breeze.—Coke which will pass through a $\frac{1}{2}$-in. square-hole screen.

NOTE.—Screening devices with round or rectangular openings are sometimes used, approximating the $\frac{1}{2}$-in. square hole in effective screening power.

Domestic Coke.—The smaller screened sizes of coke passing approximately a $2\frac{1}{2}$-in. square hole and retained on a $\frac{1}{2}$-in. square-hole screen, suitable for use in domestic stoves, heaters, etc.

NOTE.—The standard sizes are quite generally sold under the names given below and the screen sizes are average, although locally varied somewhat. Screening devices with round or rectangular openings are sometimes used, approximating the sizes of square holes given below in effective screening power:

NAME	SCREEN SIZE (SQUARE HOLE)
Egg Coke	Passing $2\frac{1}{2}$ in., Retained on $1\frac{3}{4}$ in.
Stove Coke	" $1\frac{3}{4}$ " " " $1\frac{1}{4}$ "
Nut Coke	" $1\frac{1}{4}$ " " " $\frac{3}{4}$ "
Pea Coke	" $\frac{3}{4}$ " " " $\frac{1}{2}$ "

Dry Coke.—Coke which has been dried to constant weight at temperatures not less than 104° C. nor more than 200° C. in the case of lump coke, and between 104 and 110° C. in the case of 60-mesh coke, in accordance with methods of determining mois-

ture contained in the Standard Methods of Laboratory Sampling and Analysis of Coke (Serial Designation: D 37) of the American Society for Testing Materials.[1]

Moisture.—The loss in weight sustained by heating coke in accordance with methods of determining moisture as contained in the Standard Methods of Laboratory Sampling and Analysis of Coke (Serial Designation: D 37) of the American Society for Testing Materials.[1]

Ash.—The incombustible residue remaining after ignition of coke to constant weight in an oxidizing atmosphere under conditions described under Determination of Ash contained in the Standard Methods of Laboratory Sampling and Analysis of Coke (Serial Designation: D 37) of the American Society for Testing Materials.[1]

Volatile Matter.—That part of the coal or coke which is driven off on heating in the form of tars and gases and is determined as the percentage loss in weight less the percentage of moisture sustained on heating coke in a covered platinum crucible for 7 minutes at a temperature of 950° C., in accordance with methods of determining volatile matter as contained in the Standard Methods of Laboratory Sampling and Analysis of Coke (Serial Designation: D 37) of the American Society for Testing Materials.[1]

Fixed Carbon.—The solid combustible residue remaining after heating coke in a covered platinum crucible for 7 minutes at 950° C., in accordance with methods of determining volatile matter as contained in the Standard Methods of Laboratory Sampling and Analysis of Coke (Serial Designation: D 37) of the American Society for Testing Materials.[1]

Fixed carbon = 100 per cent − (Percentage of moisture + percentage of ash + percentage of volatile matter).

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE DEFINITIONS

OF

TERMS RELATING TO COAL[1]

Serial Designation: D 142 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1922; Revised, 1924, 1925.

Proximate Analysis.—In the case of coal, determination by prescribed methods of moisture, volatile matter, fixed carbon (by difference), and ash.

Note.—Unless otherwise specified, the term "proximate analysis" does not include determinations of sulfur or phosphorus or any determinations other than those named.

Ultimate Analysis.—In the case of coal, the quantitative determination of the elementary constituents carbon, hydrogen, and oxygen (the last by difference), in the material exclusive of incombustible residue; and the quantitative determination of sulfur and nitrogen in the material as a whole.

Note 1.—Determination of phosphorus is not by definition a part of ultimate analysis of coal, but may be specified when desired.

Note 2.—When the analysis is made on an undried sample, part of the hydrogen and oxygen as determined is present in the incidental or free moisture accompanying the coal. In comparing coals on the basis of their ultimate analysis, this fact is to be given due weight.

Moisture.—That constituent part, largely made up of water, in a substance, which is separated from it in analysis under specified conditions of drying. In the case of coal, the loss incurred by heating at 105° C. in accordance with the Standard Methods of Laboratory Sampling and Analysis of Coal (Serial Designation: D 22) of the American Society for Testing Materials.[2] As occurring in coal, moisture may or may not be apparent to the eye and touch.

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[1] Criticisms of these Tentative Definitions are solicited and should be directed to Mr. W. A. Selvig, Secretary of Committee D-5 on Coal and Coke, U. S. Bureau of Mines, 4800 Forbes St., Pittsburgh, Pa.

[2] 1924 Book of A.S.T.M. Standards.

Volatile Matter.—In the case of coal, that portion (exclusive of moisture) which is driven off by destructive distillation under the conditions specified in the Standard Methods of Laboratory Sampling and Analysis of Coal (Serial Designation: D 22) of the American Society for Testing Materials.[1] It is not contained in the coal as such, but is formed by decomposition, and consists of combustible matter (tars and gases) and incombustible matter whose respective proportions vary with the type of coal.

Fixed Carbon.—In the case of coal, the solid residue, other than ash, obtained by destructive distillation under the conditions specified in the Standard Methods of Laboratory Sampling and Analysis of Coal (Serial Designation: D 22) of the American Society for Testing Materials.[1] It is made up principally of carbon, but contains also appreciable quantities of sulfur, hydrogen, nitrogen and oxygen.

Ash.—Inorganic residue remaining after complete ignition of combustible substances. In the case of coal, the residue after igniting under the conditions specified in the Standard Methods of Laboratory Sampling and Analysis of Coal (Serial Designation: D 22) of the American Society for Testing Materials.[1]

NOTE.—The ash as thus determined is less in amount than that of the actual incombustible constituents of dry coal owing to chemical changes which occur in these by burning.

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE SPECIFICATIONS

FOR

STRUCTURAL DOUGLAS FIR[1]

Serial Designation: D 23 – 20 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1916; Revised, 1920.

Material Covered. 1. (*a*) These specifications describe the properties and factors which must be considered in the production of material wherein uniformity in strength, hardness and durability are required.

(*b*) The specifications are divided into two parts:

A. Dense Douglas Fir, describing the method of selecting material for density;

B. Structural Grades, covering the production of structural timbers.

A. DENSE DOUGLAS FIR.

I. DEFINITIONS.

Definitions. 2. The following definitions are used in connection with these specifications:

(*a*) *Annual Ring.*—Each annual ring is composed of two distinct types of wood structure: namely, the porous, light-colored and light-weight spring wood formed during the first part of the growing season and the hard, dense and darker colored summer wood formed during the latter part of the growing season.

(*b*) *Summer Wood.*—Summer wood is the hard, dense portion of the annual ring. It is darker in color than the more porous spring wood.

[1] Criticisms of these Tentative Specifications are solicited and should be directed, preferably before January 1, 1926, to Mr. J. A. Newlin, Secretary of Committee D-7 on Timber, Forest Products Laboratory, Madison, Wis.

(*c*) *Dense Douglas Fir.*—This term is applied to a class of material selected with reference to the density of its wood substance content. It shall be understood that this term is descriptive of the quality of the clear wood.

II. PHYSICAL PROPERTIES.

Rate of Growth and Amount of Summerwood.

3. Dense Douglas fir shall show on one end or the other an average of at least six annual rings per inch and at least one-third summerwood measured over 3 in. on a line located as described hereinafter. Coarse-grained material, excluded by this rule, shall be acceptable provided that the amount of summerwood measured as described shall be at least one-half. Material in which the proportion of summerwood is not clearly discernible shall not be accepted.

Presence of Pith.

4. Any timber whose least dimension is less than 5 in. shall not show the pith on the inspection end; pieces whose least dimension is 5 in. or more may contain the pith.

Location of 3-in. Line.

5. (*a*) *Pith Present.*—When the least dimension is 5 in. or more, the pith being present, the line over which the rate of growth and percentage of summerwood measurements shall be made, shall run from the pith to the corner farthest from the pith. The 3-in. line shall begin at a distance from the pith equal to 2 in. less than one-half the least dimension of the piece.

When the rings are very irregular it may be necessary to shift the line somewhat around the piece to get a fair average for inspection, but the distance from the pith to the beginning of the 3-in. line must not be changed.

(*b*) *Pith not Present.*—For all pieces where the pith is not present the center of the 3-in. line shall be at the center of the end of the piece, and the direction of the 3-in. line shall be at a right angle to the annual rings.

(*c*) If a radial line of 3 in. cannot be obtained, the measurement shall be made over the entire radial line that is available.

B. STRUCTURAL GRADES.

III. DEFINITIONS.

Definitions.

6. The following definitions are used in connection with these specifications:

(*a*) *Sound and Tight Knot.*—A sound and tight knot is one which is solid across its face and which is as hard as the wood surrounding it; and is so fixed by growth or position that it will retain its place in the piece.

(*b*) *Encased Knot.*—An encased knot is one whose growth rings are not intergrown and homogeneous with the growth rings of the piece it is in. The encasement may be partial or complete; if intergrown partially or so fixed by growth or position that it will retain its place in the piece, it shall be considered a sound and tight knot.

(*c*) *Loose Knot.*—A loose knot is one not firmly held in place by growth or position.

(*d*) *Rotten Knot.*—A rotten knot is one not as hard as the wood it is in.

(*e*) *Measurement of Knots.*—In beams, the diameter of a knot on the narrow or horizontal face shall be taken as its projection on a line perpendicular to the edge of the timber. On the wide or vertical face, the smallest dimension of a knot shall be taken as its diameter.

In posts, the mean or average dimension of a knot on any face shall be taken as its diameter.

(*f*) *Ring Shake.*—A ring shake is an opening between two annual rings.

(*g*) *Round Shake.*—A round shake is a ring shake extending entirely around the perimeter of an annual ring.

(*h*) *Measurement of Shake.*—A round or ring shake shall be measured on its vertical projection.

(*i*) *Diagonal Grain* (*Including Cross and Spiral Grain*).—Diagonal grain is grain not parallel with all the edges of the piece, whether indicated by the direction of the annual rings or of the wood fibers.

IV. MANUFACTURE.

7. Structural grades shall be of sound wood and well manufactured, with square edges. **Manufacture.**

V. PHYSICAL PROPERTIES.

8. The purchaser shall list materials under their different uses, so that material of the desired requirements may be furnished. **Segregation by Uses.**

(*A*) *General Requirements.*

9. (*a*) For the limitation of knots and angle of grain a beam shall be considered as divided into three volumes as shown in Fig. 1. **Division of Beams.**

(*b*) When beams are specified by the purchaser as of two-span length, volumes 1 and 2 shall be considered as extending

between points located one-eighth the length of the beam from each end.

Knots.

10. Structural timber shall be free from rotten or unsound knots or knots in groups or clusters. Sound and tight knots, limited in size and position as hereinafter provided, will be permitted.

Angle of Grain.

11. (*a*) Beams shall not have diagonal or spiral grain in volumes 1 and 2 with slope greater than 1 in 20.

(*b*) Posts shall not have diagonal or spiral grain with slope greater than 1 in 15, unless otherwise specified by the purchaser.[1]

Shakes and Checks.

12. Round or ring shakes shall not occupy more than one-fourth the least dimension of either end of a timber. Any combination of checks or shakes which reduce the strength to a greater extent than the allowable round shakes will not be permitted. Shakes shall not show on any face of a timber.

Tension Members.

13. Defects in timber listed as tension members shall be limited as in beams, unless specified by the purchaser to be

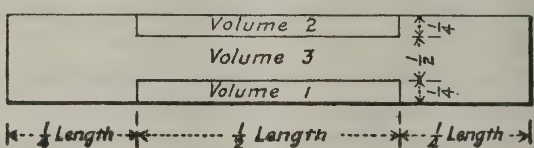


FIG. 1.

limited throughout the entire stick as in volumes 1 and 2 of beams.[2]

Heart Requirement in Bridge and Trestle Timbers.

14. If specified by the purchaser, bridge and trestle timbers shall have the following heart requirements.

(*a*) Beams, stringers, girders and deep joists, caps, sills and posts shall show not less than 85 per cent of heart on each of the four sides, measured across the sides anywhere in the length of the piece.

(*b*) Longitudinal struts or girts shall show all heart on one face; the other face and two sides shall show not less than 85 per cent of heart, measured across the face or side anywhere in the length of the piece.

(*c*) Longitudinal cross braces, sash braces and sway braces shall show not less than 85 per cent of heart on two faces.

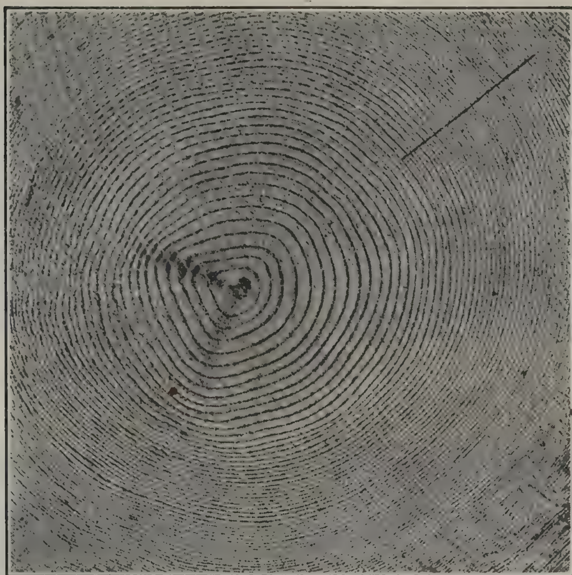
(*B*) *No. 1 Structural.*

Density and General Requirements.

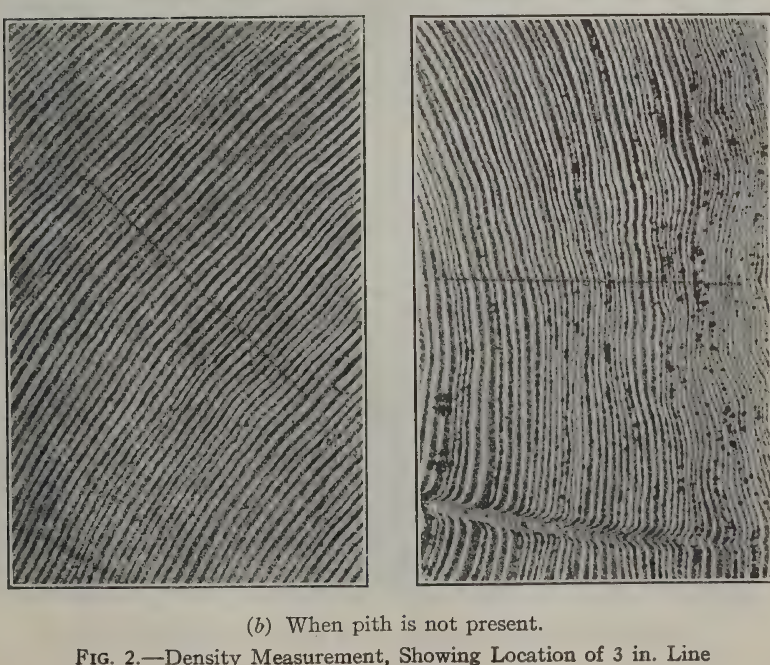
15. No. 1 structural Douglas fir timbers shall be of dense Douglas fir and shall meet the preceding General Requirements.

[1] See Appendix, Section 4.

[2] See Appendix, Section 2.



(*a*) When pith is present.



(*b*) When pith is not present.

FIG. 2.—Density Measurement, Showing Location of 3 in. Line

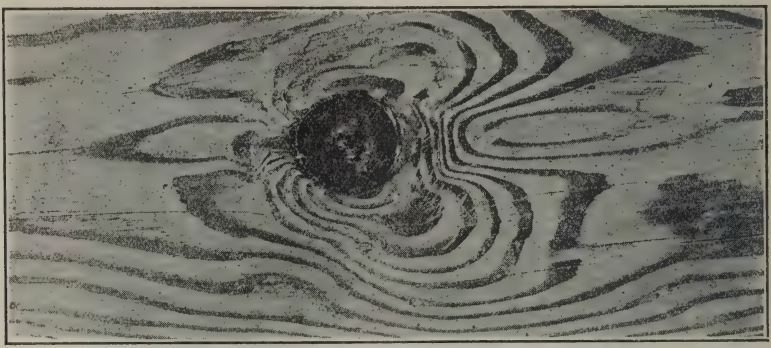


Fig. 3.—A Round Knot.

Fig. 4.—Measurement of a Knot, Measurement Perpendicular to the Edge of the Timber.

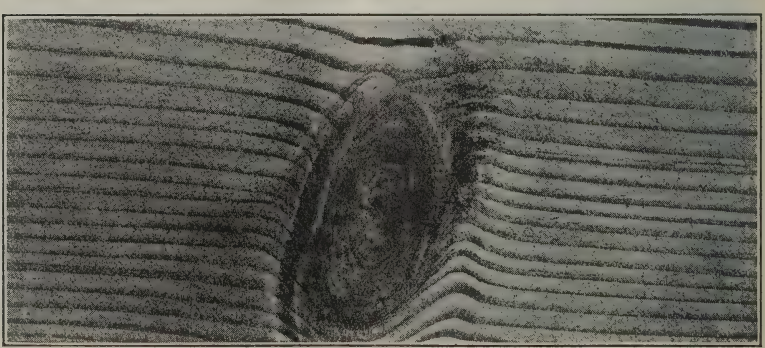


Fig. 5.—Measurement of a Knot on its Smallest Diameter.

16. They may have pitch pockets not over 6 in. long or $\frac{3}{8}$ in. wide and wane not to exceed 1 in. on one corner nor one-sixth the length of the piece. **Pitch Pockets and Wane.**

17. (*a*) *Loose Knots.*—Loose knots larger than $\frac{1}{2}$ in. shall not be permitted. **Knots.**

(*b*) *Beams, Stringers, Girders and Deep Joists.*—Beams, stringers, girders and deep joists shall not have in volumes 1 and 2 knots greater in diameter than one-fourth the width of the face in which they occur with a maximum of $1\frac{1}{2}$ in. in diameter. They shall not have in volume 3, on either end of the center half of a beam, knots larger than one-fourth the width of the face in which they occur. Knots within the center half of the length of a beam shall not exceed in the aggregate the width of the face in which they occur.

FIG. 6.—Measurement of a Spike Knot, Measurement Perpendicular to the Edge of the Timber.

(*c*) *Caps, Sills and Posts.*—Caps, sills and posts shall be free from knots larger than one-fourth the width of the face in which they occur, with a maximum of 3 in. in diameter.

(*d*) *Longitudinal Struts or Girts.*—Longitudinal struts or girts shall be free from knots over 2 in. in diameter.

(*e*) *Longitudinal Cross Braces, Sash Braces and Sway Braces.*—Longitudinal cross braces, sash braces and sway braces shall be free from knots larger than one-third the width of the face in which they occur, with a maximum of 2 in. in diameter.

(C) No. 2 Structural.

General Requirements. 18. No. 2 structural timbers shall meet the preceding General Requirements and shall include timbers not passing the No. 1 grade because of having:

(*a*) Less density than is required; or

(*b*) greater defects than are permitted.

Pitch Pockets and Wane. 19. They may have pitch pockets not longer than 12 in. or wider than $\frac{3}{8}$ in. and may have 2 in. wane on one corner or the equivalent on two or more corners of 10 by 10-in. timbers, with wane in proportion on smaller or larger sizes.

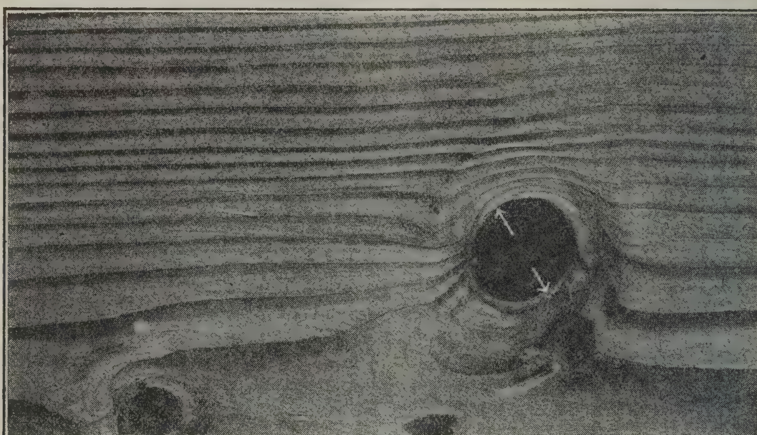


FIG. 7.—Measurement of the Mean Diameter of a Knot.

Knots. 20. (*a*) *Loose Knots.*—Loose knots larger than one-half the size of the knots allowed shall not be permitted.

(*b*) *Beams, Stringers, Girders and Deep Joists.*—Beams, stringers, girders and deep joists shall not have in volumes 1 and 2 knots greater in diameter than as follows:

(1) If of dense Douglas fir, one-third the width of the face in which they occur with a maximum of 3 in.

(2) If not of dense Douglas fir, one-fourth the width of the face in which they occur with a maximum of $1\frac{1}{2}$ in.

They shall not have in volume 3, on either end of the center half of the beam, knots larger than one-third the width of the face in which they occur. They shall not have loose knots in volume 3 larger than $1\frac{1}{2}$ in. Knots within the center half of the length of a beam shall not exceed in the aggregate twice the width of the face in which they occur.

(*c*) *Caps and Sills.*—Caps and sills shall be free from knots larger than one-half the width of the face in which they occur with a maximum of 3 in. in diameter.

(*d*) *Posts.*—Posts shall be free from knots greater in diameter than as follows:

(1) If of dense Douglas fir, one-third the least dimension of the post with a maximum of 4 in.

(2) If not of dense Douglas fir, one-fourth the least dimension of the post with a maximum of 3 in.

(*e*) *Longitudinal Struts or Girts.*—Longitudinal struts or girts shall be free from knots over 2 in. in diameter.

(*f*) *Longitudinal Cross Braces, Sash Braces and Sway Braces.*—Longitudinal cross braces, sash braces and sway braces shall be free from knots larger than one-third the width of the face in which they occur with a maximum of 2 in. in diameter.

VI. STANDARD SIZES.

21. Rough timbers shall be sawed to standard size and when green shall not be more than $\frac{1}{4}$ in. less than the nominal size specified. **Standard Size Rough.**

22. When timbers 4 by 4 in. or larger are ordered sized, they may when green be $\frac{1}{2}$ in. less than the nominal size specified, either S1S1E or S4S, unless otherwise specified. **Standard Size Green.**

APPENDIX.

The following information as to working stresses and the use and application of the Specifications for Structural Douglas Fir is important to a proper understanding of the specifications and is given below in the form of an Appendix to the Specifications. The appendix is not, however, a part of the specifications.

WORKING STRESSES.

1. The following working stresses are based upon recommendations by the Forest Products Laboratory, U. S. Forest Service, for Structural Douglas Fir:

	No. 1 Structural.	No. 2 Structural.[1]
Bending: Extreme Fiber Stress[2]		
Wet Location	1 100 lb. per sq. in.	900 lb. per sq. in.
Outside Location	1 400 " " "	1 100 " " "
Dry Location	1 600 " " "	1 300 " " "

[1] In this grade pieces of exceptionally low density should not be placed in positions where they will receive long-continued loads producing maximum allowable stresses.

[2] Where timbers are in direct tension the same values as for extreme fiber stress in bending may be used. Wood has greater resistance to tension than to any other kind of stress and it has been found difficult to break it in a true tension test.

	No. 1 Structural.	No. 2 Structural.
Maximum Shear:		
Horizontal, All Locations[1]...	100 lb. per sq. in.	90 lb. per sq. in.
Compression:		
Parallel to Grain[2]		
Wet Location............	900 " " "	800 " " "
Outside Location.........	1 100 " " "	900 " " "
Dry Location............	1 200 " " "	1 000 " " "
Perpendicular to Grain		
Wet Location............	225 " " "	200 " " "
Outside Location.........	250 " " "	225 " " "
Dry Location............	350 " " "	300 " " "
Elasticity:		
Average Modulus, All Locations[3]..............	1 600 000 " " "	1 400 000 " " "

USE OF THE DENSITY AND STRUCTURAL GRADING RULES.

2. When timbers to be used in direct tension are designed for full working stresses, defects throughout the entire stick should be limited as in volumes 1 and 2 of beams.

3. When beams are of two-span length, volumes 1 and 2 should be considered as extending between points located one-eighth the length of the beam from each end.

4. When posts are designed for less than full working stresses, the angle of grain may be increased proportionately to the reduction in stress; that is, with a working stress one-half the full working stress allowed, the angle of grain may be 1 in $7\frac{1}{2}$.

5. Dense material may be obtained in any commercial grade and independently of structural properties; density should be required where strength or resistance to abrasion or decay are factors.

6. For sills, caps, plates and other members where bending stresses are low compared with compression across the grain, density should be required but special limitation as to sound knots and angle of grain is not necessary.

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[1] These values are based on present inadequate information concerning the effect of seasoning on shearing values in Douglas fir, further investigation in regard to which is now under way.

[2] A short column is more likely to fail at the end than at any other point and the variations in moisture are greater there; for these reasons the same stresses should be used in end bearing as for short columns. For "long columns," that is, compression members with an unsupported length greater than about ten times their least diameter, these values should be reduced according to a column formula. Such formulas are given and discussed in the various text-books on mechanics, and in treatises on structural design.

[3] Timber constantly yields under long-continued heavy loading, and will fail finally at a load little more than half that required to break it when applied rapidly in a test; for application to a beam which is to be subjected to long-continued loading, therefore, moduli of elasticity one-half those given above should be used.

7. For posts, where large sizes are required for harmony with the balance of the construction and where resistance to decay is not an important factor, timbers without density requirement may be used.

8. For studding and wall construction, where the spacing of members is governed by requirements for nailing or supports to wall covering, commercial grades without density requirement may be used.

9. For joists, where stiffness is a controlling factor rather than bending stresses or resistance to decay, density need not be required, there being only slightly greater stiffness in dense material.

10. Heartwood and sapwood are of equal strength but when durability is a factor, as in exposure to decay-producing conditions, sapwood should be limited or all heartwood be required.

Inspection of Structural Grades.

11. The rules for structural grades contain minimum requirements and maximum defects, all of which may be present at one time; when a particular timber is slightly below the provisions of the grade in some of these properties and somewhat above in others the relative effect should be considered in accepting or rejecting it.

Reinspection of Dense Douglas Fir.

12. No arbitrary rules for the inspection of lumber can be maintained with satisfaction. Inspection of lumber is not an exact science, and a reasonable variation of opinion between inspectors should be recognized.

13. Upon reinspection of dense Douglas fir, all timbers having one-third or more summerwood shall be accepted as of grade. For every three timbers thus accepted, one of the remaining timbers having between 30 and $33\frac{1}{3}$ per cent summerwood shall also be included.

14. Any remaining material having more than 30 per cent summerwood, and also all material found to have less than 30 per cent, shall be received as not of grade, with settlement subject to agreement between shipper and purchaser.

TENTATIVE METHODS OF TESTING SMALL CLEAR SPECIMENS OF TIMBER[1]

Serial Designation: D 143 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1924.

The everyday use of timber for multitudinous purposes makes manifest a continual need of data on its mechanical properties. The great variety of species, the variability of the material, the continually changing conditions of supply, the many factors affecting test results, all combine to make the technique of testing wood unique in its complexity.

In the preparation of this procedure for testing small clear specimens, consideration was given both to the desirability of adopting methods which would yield results comparable to those already available and to the possibility of embodying such improvements as experience has shown desirable. In view of the many thousands of tests made under a single comprehensive plan by the U. S. Forest Service, the Forestry Branch of the Department of the Interior, Canada, and other similar organizations, the procedure naturally conforms closely to the methods used by these institutions. This procedure is the outgrowth of a study of both American and European experience and methods. Its general adoption will tend toward a world-wide unification of results, permitting an interchange and correlation of data, and will establish the basis for a cumulative body of fundamental information on the timber species of the world.

The methods under consideration represent the entire procedure from selection of the trees to the manipulation of the test, thus controlling factors, such as the size and proportion of test specimens and rate of loading, which may influence results. No attempt has been made to cover methods of computation and analysis, as these questions may be considered independently at any time. Such sample data and computation sheets and cards have been incorporated, however, as were thought to be of assistance to the investigator in systematizing records.

Purpose of Tests.

1. Tests on small clear specimens of wood are made to afford:

(*a*) Data for comparing the mechanical properties of various species.

[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. J. A. Newlin, Secretary of Committee D-7 on Timber, Forest Products Laboratory, Madison, Wis.

(*b*) Data for the establishment of correct strength functions which, in conjunction with results of tests of timbers in structural sizes,[1] afford the basis for fixing allowable stresses.

(*c*) Data upon which to determine the influence on the mechanical properties of such factors as density, locality of growth, position in cross-section, height of timber in the tree, change of properties with seasoning, and change from sapwood to heartwood.

Kind of Tests.

2. The principal mechanical tests are static bending, compression parallel to grain, impact bending, compression perpendicular to grain, hardness, shear parallel to grain,[2] tension perpendicular to grain, cleavage, and tension parallel to grain. These tests are made on both green and air-dry material as hereinafter specified. In addition, such physical properties as specific gravity, radial shrinkage, tangential shrinkage, and shrinkage in volume are determined.

MATERIAL.

I. COLLECTION.

(*A*) *Selection.*

Authentic Identification.

3. The material shall be from trees selected in the forest by one qualified to indentify the species.

Selection and Number of Trees.

4. For each species to be tested from a given locality, at least five representative trees of merchantable size and of approximately average age shall be selected.

Selection and Number of Bolts.

5. The material from five trees of each species from a given locality shall be selected as follows:

(*a*) From one tree, the entire merchantable bole, if not over 16 ft. in length;[3] if over 16 ft. in length, four 4-ft. sections or bolts, the first to be taken at the stump, the second and third at distances of one-eighth and three-eighths, respectively, of the merchantable length above the stump or as near thereto as possible; and the fourth at the top of the merchantable length.

(*b*) From two trees, the 8-ft. section next above the 8-ft. butt log.

(*c*) From two trees, the 4-ft. section next above the 12-ft. butt log.

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[1] See Tentative Methods of Conducting Static Tests of Timbers in Structural Sizes (Serial Designation: D 198 - 24 T) of the American Society for Testing Materials, p. 565.

[2] The test for shearing strength perpendicular to the grain (sometimes termed "vertical shear") is not included as one of the principal mechanical tests since in such a test the strength is limited by the shearing resistance parallel to the grain.

[3] This tree is intended to furnish test material for studying, among other things, the variation of properties with height in trees and provides for this purpose a 4-ft. section from different heights. Experience has shown that four sections or bolts, each 4 ft. in length, will usually be sufficient for studying the influence of height in tree on the mechanical properties.

Selection from Small Trees.

6. In cases where the logs or bolts are over 36 in. in diameter, a single flitch 6 in. in thickness, taken through the pith in a north and south direction and representing the full diameter of the log, may be substituted, in the same length, for the full log or bolt specified in Section 5.

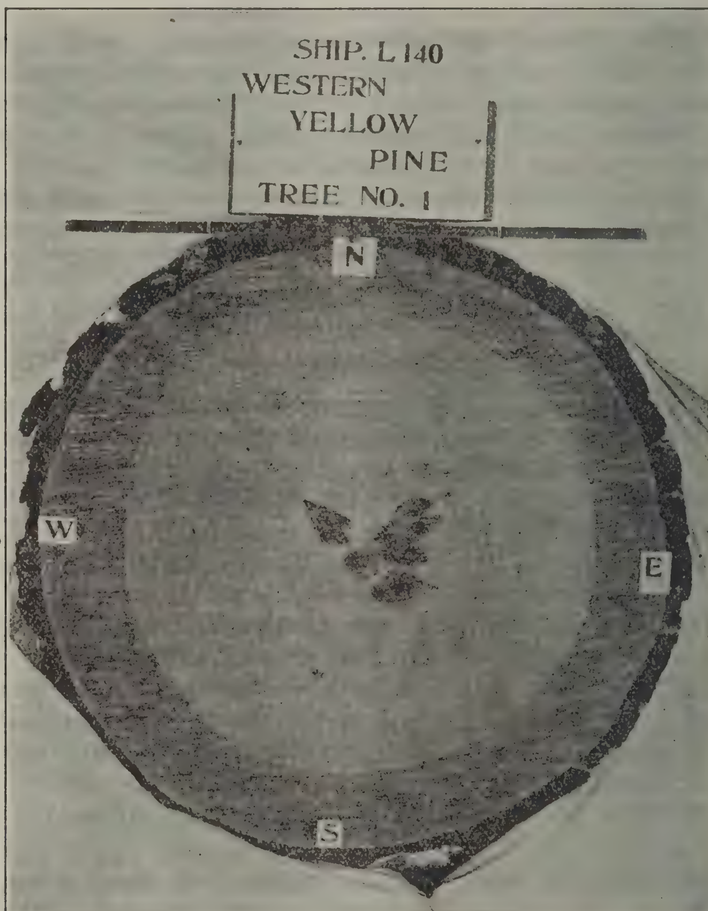

FIG. 1.—Section of Log Selected for Test Material.

Substitution of Flitches for Bolts.

7. When the trees are relatively small in diameter, material in addition to that provided for in Sections 5 and 6 shall be collected, whenever possible, to insure a sufficient number of tests to give a good average for the species.

(*B*) *Field Marking.*

8. Each tree shall be given an arabic number, the numbering in any given shipment to be consecutive for trees of a given species. **Tree Designation.**

9. Each 4 ft. of length of a tree or log shall be considered a "bolt." Bolts shall be designated by small letters, beginning with *a* for the 4-ft. section next above the stump. Bolt letters, therefore, indicate position with respect to height in tree. **Bolt Designation.**

10. The tree number and bolt designation shall be plainly marked upon each log selected by the collector. Thus the 16-ft. butt log of Tree No. 2 would be designated *2abcd*. Steel dies are recommended for marking the butt end of the logs. **Manner of Marking.**

11. The north side of each log shall be indicated in some convenient manner. **Cardinal Point Indicated.**

12. All material collected from a given locality and shipped at one time shall be given a shipment number or other designation. **Shipment Number.**

TABLE I.—SHIPMENT DESCRIPTION FIELD NOTES.

Project No., *124* Shipment No., *323*
Tree No., *10* Species, *Lodgepole pine*
Locality cut: State, *Montana* County, *Jefferson*
Township, *6 N., R. 7 W., M. P. M.* Date cut, *Oct. 10, 1914*
Slope, *Moderate* Absolute elevation, *6600 ft.* Aspect, *southeasterly*
Undergrowth, *None* Soil, *Deep, fresh-decomposed granite*
Height, *73.9 ft.* Seedling or sprout, *Seedling* Age, *140*
Crown, *Length 25 ft., width 12 ft.* D.B.H. *11.1 in.*
Stem, *Sound, smooth, clear, length 20 ft.* Date sawed, *Oct. 10, 1914*
How and when transported from woods, *By sleigh and wagon, Oct. 13 to 15, 1914.*

LIST OF SPECIMENS SAWED.

Log No.	Bolts.	Length, ft.	Diameter Inside Bark.		Volume.	Feetage, b. m.	Weight, Oct. 15, lb.
			Butt, in.	Top, in.			
1	bcd	12.1	11	9.6	..	..	336
2	efg	12.1	9.6	8.9	..	..	289
3	hij	12.1	8.9	7.7	..	..	242
4	klm	12.1	7.7	6.1	..	..	176
5	nop	12.1	6.1	3.1	..	..	95

REMARKS:[1] *Stump height, 1.0 ft. Butt off on account of frost crack 3.0 ft. Length of top above last section taken, 9.4 ft. Total age, 140 years. Tree was dominant throughout life. Rate of growth, regular and good average for Site I. Lower slope type, although of more than average height for the age and diameter found on poorer sites*

(*C*) *Field Descriptions.*

13. (*a*) Complete field notes describing the material shall be fully and carefully made by the collector. These notes shall in general, supply data as outlined in Table I. **Field Description.**

(*b*) Photographs of the standing trees selected shall be taken.

(*D*) *Preparation for Shipment.*

Preparation for Shipment. 14. (*a*) The bark will be left on each log, and care shall be taken to keep the bark intact. The ends of the logs shall be carefully painted to retard or prevent end drying and end checking.

(*b*) Record shall be made of the shipment routing, bill of lading,

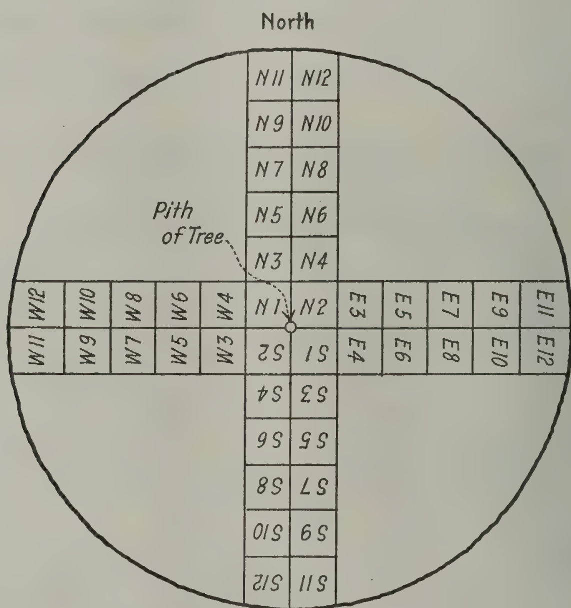

FIG. 2.—Sketch Showing Method of Cutting up the Bolt and Marking the Sticks.

kind of shipment, date of shipment, and condition of material when shipped.

(*c*) Record shall also be made of date of receipt of shipment at destination, its condition and method of storage.

II. DISPOSITION.

(*A*) *Storage of Logs at Destination.*

Storage of Logs. 15. Material shall not be kept in the bolt or log form long enough to permit damage by checks, decay, stains, or insect attack. The

logs shall be piled on skids, free from contact with the soil, and shall not be stored where subjected to artificial heat. In addition they should preferably be protected from the sun.

(B) *Photographing, Sawing and Final Marking.*

16. The top end of each *d* or *c-d* bolt shall be photographed. It is suggested that a rule be so placed on the log as to indicate the scale of the photograph and that the cardinal points be indicated on the cross-section. Fig. 1 shows a photograph of this kind. **Photographing Ends of Bolts.**

FIG. 3.—Cross-Sections of Bending Specimens Showing Different Rates of Growth of Longleaf Pine (2 by 2-in. Specimens)

17. (*a*) All bolts shall be marked on the top end into $2\frac{1}{2}$ by $2\frac{1}{2}$-in. squares as shown in Fig. 2, and sawed into nominal $2\frac{1}{2}$ by $2\frac{1}{2}$-in. sticks. The letters *N*, *E*, *S*, and *W* indicate the cardinal points. **Sawing of Bolts.**

(*b*) When flitches are substituted for bolts (Section 6), the same general marking and numbering scheme of Paragraph (*a*) shall be followed in so far as it is applicable.

18. All test sticks shall bear the shipment number, the tree number, stick number, and bolt designation, to be known respectively **Marking of Test Sticks.**

as Shipment No., Piece No., Stick No., and mark. Thus 400-1-*N*4*d* represents Stick *N*4 of Bolt *d*, Tree 1, Shipment 400.

(*C*) *Matching for Air-dry Tests.*

Composition Bolts.

19. In the tests made to determine the effect of air-drying on strength, the sticks from the *c* bolt shall be interchanged with those of the *d* bolt of the same tree to form two composition bolts, each composition bolt being complete and being made of equal portions of *c* and *d* bolts.[1] The sticks from one of these composition bolts shall be tested green and those from the other shall be tested after air drying. The sticks tested either green or air dry shall be regarded as if they were from the same bolt.

Schedule for Forming Composition Bolts.

20. The division of green and air-dry sticks shall be made according to the following schedule in which the numbers refer to stick numbers:

Composition Bolt to be Tested Green.—

Lower bolt *c*....................	1	4, 5	8, 9	
Upper bolt *d*........................	2, 3	6, 7		10, etc.

Composition Bolt to be Air Dried and Tested.—

Lower bolt *c*........................	2, 3	6, 7		10, etc.
Upper bolt *d*....................	1	4, 5	8, 9	

(*D*) *Disposition of Sticks.*

Green Material.

21. The sticks ($2\frac{1}{2}$ by $2\frac{1}{2}$ in. by 4 ft.) to be tested green shall be kept in an unseasoned condition while awaiting preparation for test by being stored in a framed pit or other suitable container, where they shall be close piled and covered with damp sawdust or in some other suitable manner. As material is required for test, it shall be removed from this pit, or container, surfaced on all four sides to 2-in. by 2-in. in cross-section, sawed to test size and kept covered with a damp cloth, in an air-tight container at a temperature of about 70° F. (as nearly as practicable) until the time of test. Care shall be taken to avoid as much as possible the storage of green material in any form. Sticks to be tested in a green condition should usually not be sawed from the log form in quantities greater than is required to meet the

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[1] The 8-ft. section (*c* and *d* bolts) from each of the two trees provided for under Section 5 (*b*) is intended for tests to determine the effect of air drying on strength.

testing demands for from a few days to not more than two weeks, depending on the prevailing conditions.

22. (*a*) The ends of the sticks to be air dried ($2\frac{1}{2}$ by $2\frac{1}{2}$ in. by 4 ft.) shall be dipped in melted paraffin or other substance suitable to retard checking. The material shall be piled so as to have a space of at least $\frac{1}{2}$ in. on each side of each stick to permit circulation of air. The material shall be stored in a place allowing free access of air, but protected from sunshine, rain, snow, and moisture from the ground. The sticks in drying shall not be subjected to artificial heat. **Air-dry Material.**

(*b*) All of the sticks from each composite bolt to be air dried shall be weighed when stored and at sufficiently frequent intervals thereafter to get accurate data on the progress of seasoning. No

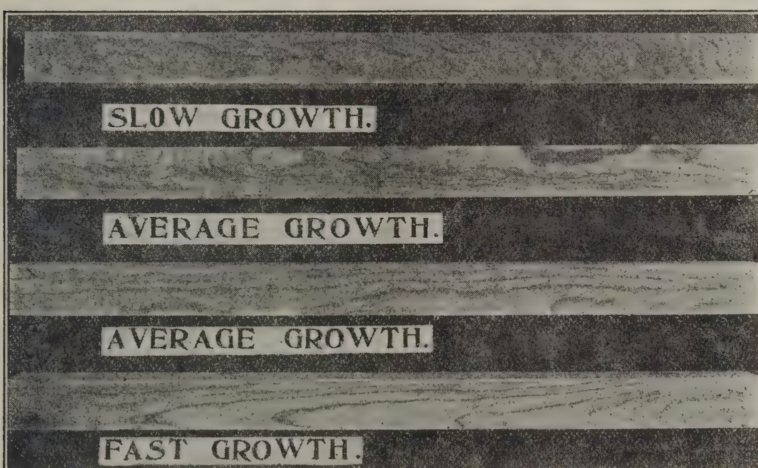

FIG. 4.—Tangential Surfaces of Bending Specimens of Different Rates of Growth of Jeffrey Pine (2 by 2 by 30-in. Specimens).

material shall be considered thoroughly air dried and properly conditioned for testing until practically constant weight has been reached. (Wood absorbs and gives off moisture with changing atmospheric conditions, consequently it never comes to absolutely constant weight.)

(*c*) When the material has reached equilibrium, moisture sections 1 in. in length shall be taken from about 10 per cent of the sticks to determine the actual moisture content. These moisture specimens shall be cut not less than 1 ft. from the ends of the sticks, and in such a way as to prevent any appreciable loss of material for testing. When thoroughly air dry, the material shall be surfaced on four sides to 2 in. by 2 in. in cross-section and tested.

TESTING.

III. ORDER, SELECTION, AND NUMBER.

(*A*) *Order.*

Order of Tests.

23. The order of tests in all cases shall be such as to eliminate as far as possible from the comparisons the effect of changes in the specimen due to such factors as storage and weather conditions.

(*B*) *Selection.*

Preference in Selecting Specimens.

24. In case the material from a given bolt should be insufficient to furnish all the test specimens hereinafter required, the preferential order of mechanical tests to be used in selecting specimens shall be as follows: Static bending, compression parallel to grain, impact bending, compression perpendicular to grain, hardness, shear parallel to grain, tension perpendicular to grain, cleavage, and tension parallel to grain.

Test Pieces from Bending Specimens After Failure.

25. In many cases where a shortage of material exists, certain test specimens may be taken from the uninjured portion of the static and impact bending specimens remaining after test, provided proper care is used in the selection.

Quality of Test Material.

26. Only clear straight-grained material, free from decay and other defects, shall be used for the tests. However, small knots and other similar defects may be admitted in such specimens as static bending when their location is such that it is certain they will not in any way influence the failure or otherwise affect the strength of the specimen.

(*C*) *Number of Tests for Each Bolt.*

Static Bending.

27. One static bending specimen shall be taken from each pair of sticks. A pair consists of two adjacent sticks equi-distant from the pith, as *W*3 and *W*4, Fig. 2. In the composite bolts tested to afford a comparison of the strength of green and air-dry material, the pair of sticks shall be constituted as above, except that the sticks in this case will be from different bolts. Thus *W3d* and *W4c* constitute one pair of sticks to be tested green, and *W3c* and *W4d* the corresponding pair to be tested air dry (Section 20).

Compression Parallel to Grain.

28. One compression-parallel-to-grain specimen shall be taken from each stick. Load-compression curves shall be taken on 20 per cent of the specimens.

Impact Bending.

29. Eight impact-bending specimens shall be taken from each bolt, selection being made from the sticks remaining after obtaining the static bending tests. Two of the specimens shall be selected from

near the pith, two from near the periphery, and four which are representative of the cross-section.

30. One compression-perpendicular-to-grain specimen shall be taken from each of 50 per cent of the sticks selected for static bending. **Compression Perpendicular to Grain.**

31. One hardness specimen shall be taken from each of the other 50 per cent of the static-bending sticks. **Hardness.**

32. Eight shear-parallel-to-grain specimens shall be selected from the unused portion or ends of four sticks from which bending specimens have been selected. Two specimens shall be taken from near the pith, two from near the periphery, and four that are representative of the average growth of the cross-section of the bolt. One of each pair of specimens from the same stick shall be tested in radial shear (surface of failure radial) and the other in tangential shear (surface of failure tangential). **Shear Parallel to Grain.**

33. Eight tension-perpendicular-to-grain specimens shall be selected from four sticks in a manner similar to that for shear (Section 32). One of each pair of specimens from the same stick shall be tested in radial tension (surface of failure radial) and the other in tangential tension (surface of failure tangential). **Tension Perpendicular to Grain.**

34. Eight cleavage specimens shall be selected from four sticks in a manner similar to that for shear (Section 32). One of each pair of specimens from the same stick shall be tested in radial cleavage)surface of failure radial) and the other in tangential cleavage (surface of failure tangential). **Cleavage Perpendicular to Grain.**

35. Four tension-parallel-to-grain specimens shall be chosen of which one shall be selected from near the pith, one from near the periphery and two which are representative of the cross-section. **Tension Parallel to Grain.**

36. Six specific gravity and shrinkage-in-volume specimens shall be selected from the unused portion of bending or tension-parallel-to-grain sticks, selected so as to give one from near the pith, one from near the periphery, and four that are representative of the average growth of the cross-section of the bolt. These specimens shall be selected only from the sticks to be tested in a green condition. **Specific Gravity and Shrinkage in Volume.**

37. Two radical shrinkage specimens shall be obtained from each *d* bolt. They shall be cut from the "sectors" or "quadrants" remaining after sawing (Fig. 2) or from disks cut from near the end of the bolt. When a disk is used, care must be taken to see that it is green, and has not been affected by shrinking and checking which is common near the end of the bolt. The specimens shall not be surfaced. Radial shrinkage specimens shall be cut with their width in the radial direction. One shall be taken from the heartwood, and the other from near the periphery. When possible, the second shall consist entirely of sapwood. **Radial Shrinkage.**

Tangential Shrinkage.

38. Two tangential shrinkage specimens shall be obtained from each *d* bolt. They shall be selected at the same time and in a manner similar to radial-shrinkage specimens (Section 37), except the width shall be in a tangential direction. The specimens shall not be surfaced. One shall be taken from the heartwood, the other from near the periphery and when possible shall consist entirely of sapwood. The heartwood and the sapwood specimens shall be taken adjacent to the respective specimens selected for radial shrinkage.

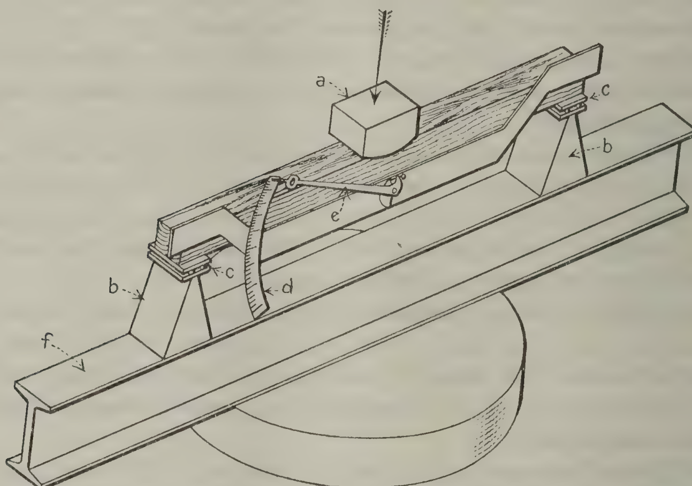

FIG. 5.—Diagrammatic Sketch of Method of Conducting Static Bending Test.

IV. GENERAL INSTRUCTIONS.

(*A*) *Photographs of Sticks.*

Sticks to be Photographed.

39. Four of the bending sticks from each species shall be selected for photographing, as follows: 2 average growth, 1 fast growth, and 1 slow growth. These sticks shall be photographed in cross-section and on the radial and tangential surfaces. Fig. 3 is a typical photograph of the cross-section of test specimens and Fig. 4 of the tangential surface of bending specimens.

(*B*) *Control of Moisture Content.*

Control of Moisture Content.

40. As previously specified (Section 22) sticks for test in the air-dry condition shall be brought to practically constant weight

before test. Tests shall then be carried out in such a manner that large changes in moisture content will not occur. (For instance, in ordinary heated rooms the relative humidity is usually below 30 per cent. If wood which has been dried to practically constant

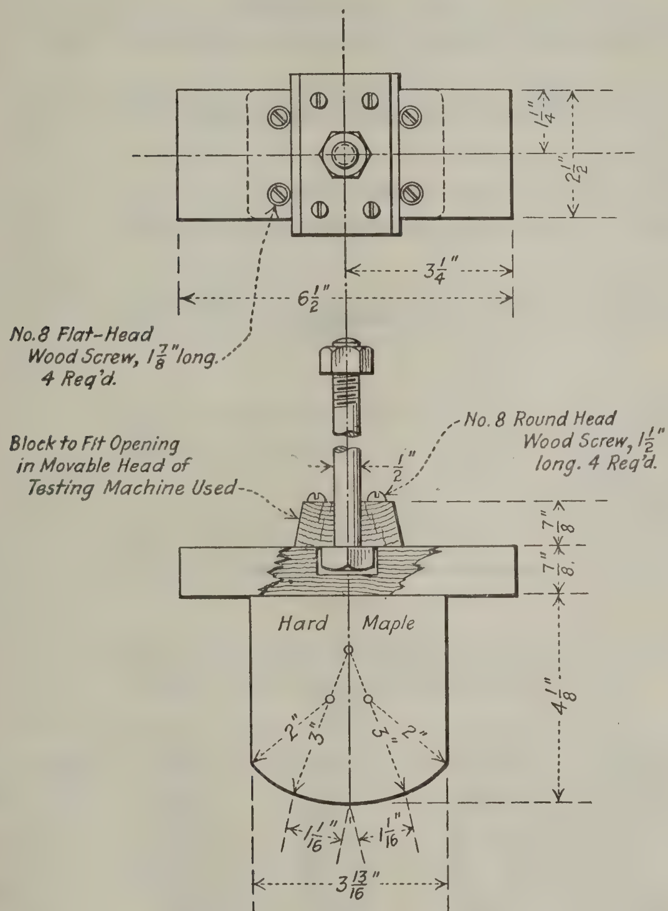

FIG. 6.—Details of Bearing Block for Bending Tests.

weight under the condition specified by Section 22 is brought into such a room, a considerable and rather rapid drying out will occur.) To prevent such changes, the testing room and rooms for the preparation of test specimens should preferably have some means of humidity control.

(C) *Record of Heartwood and Sapwood.*

Proportion of Sapwood. 41. The estimated proportion of sapwood present shall be recorded for each test specimen.

V. PROCEDURE.

(A) *Static Bending.*

Size of Specimens. 42. The static bending tests shall be made on nominal 2 by 2

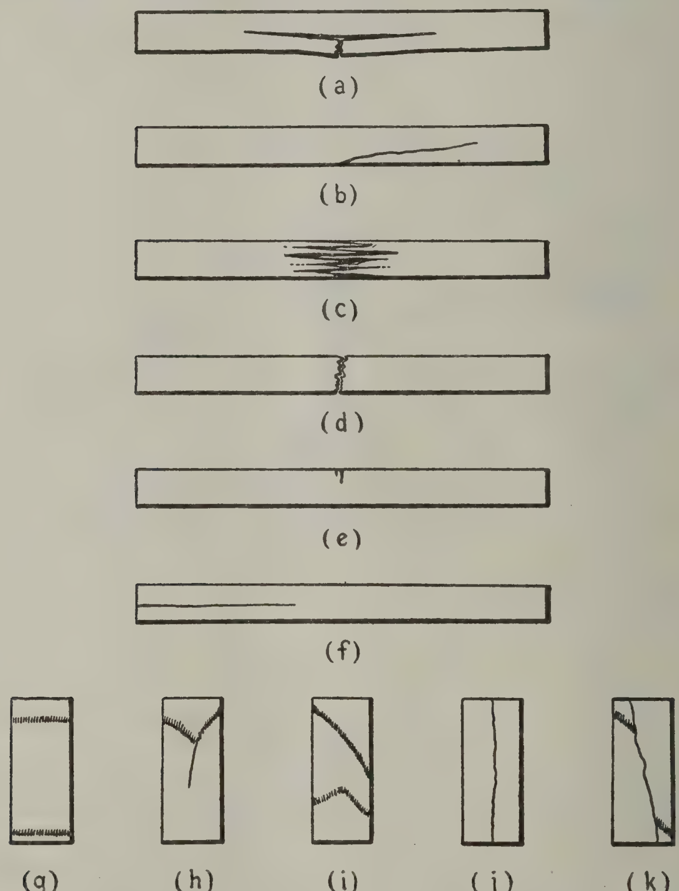

Fig. 7.—Types of Failures: *a* to *f*, Bending; *g* to *k*, Compression Parallel to Grain.

by 30-in. specimens. The actual height and width at the center, and the length shall be measured.

Loading Span and Supports. 43. Center loading and a span length of 28 in. shall be used. Both supporting knife edges shall be provided with bearing plates and rollers having a total thickness of approximately $1\frac{1}{4}$ in. in order to

bring the neutral plane of the test specimen about $2\frac{1}{4}$ in. from the knife edges (Fig. 5.)

Bearing Block. 44. A bearing block of the form and size of that shown in Fig. 6 shall be used for applying the load.

Placement of Growth Rings. 45. The specimen shall be placed so that the load will be applied through the bearing block to the tangential (flat-sawn) surface nearest the pith.

Rate of Application of Load. 46. The load shall be applied continuously throughout the test at a rate of 0.10 in. per minute.

Load-Deflection Curves. 47. (*a*) Load-deflection curves shall be taken to or beyond the maximum load for all static bending tests. In one-third of the tests, the curves shall be continued to a 6-in. deflection, or until the specimen fails to support a load of 200 lb.

(*b*) Deflections of the neutral plane at the center of the length shall be taken with respect to points in the neutral plane above the supports.

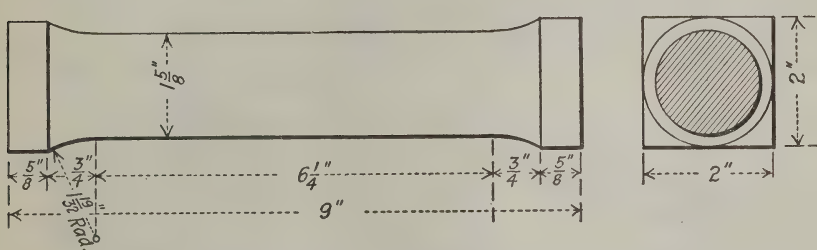

FIG. 8.—Compression-Parallel-to-Grain Test Specimen Special Form.

(*c*) Within the elastic limit, deflection readings shall be taken to 0.0025 in. After the elastic limit has been reached, less refinement is necessary in observing deflections, and these may then be read directly to the nearest 0.01 in. by means of a wire stretched across a scale. Fig. 5 illustrates the method of test and type of deflectometer recommended for deflections up to 1 in.

(*d*) The load and deflection of first failure, the maximum load, and points of sudden change shall be shown on the curve sheet[1] although they may not occur at one of the regular load or deflection increments.

Description of Failures. 48. Bending failures shall be classified according to the appearance of the fractured surface and according to the manner in which the failure develops. The fractured surfaces may be roughly divided into brash and fibrous, the term "brash" indicating an abrupt failure

[1] See Fig. 1 of the Appendix for a sample static bending data sheet form. Fig. 2 of the Appendix shows a sample computation data card.

and the term "fibrous" indicating a fracture showing splinters. The test failures shall be classified and described as follows:

Simple tension (see Fig. 7*a*);
Cross grain[1] tension (see Fig. 7*b*);

The presence of cross grain having a slope which deviates more than 1 in 20 from the longitudinal edges of the specimen shall be cause for culling the test.

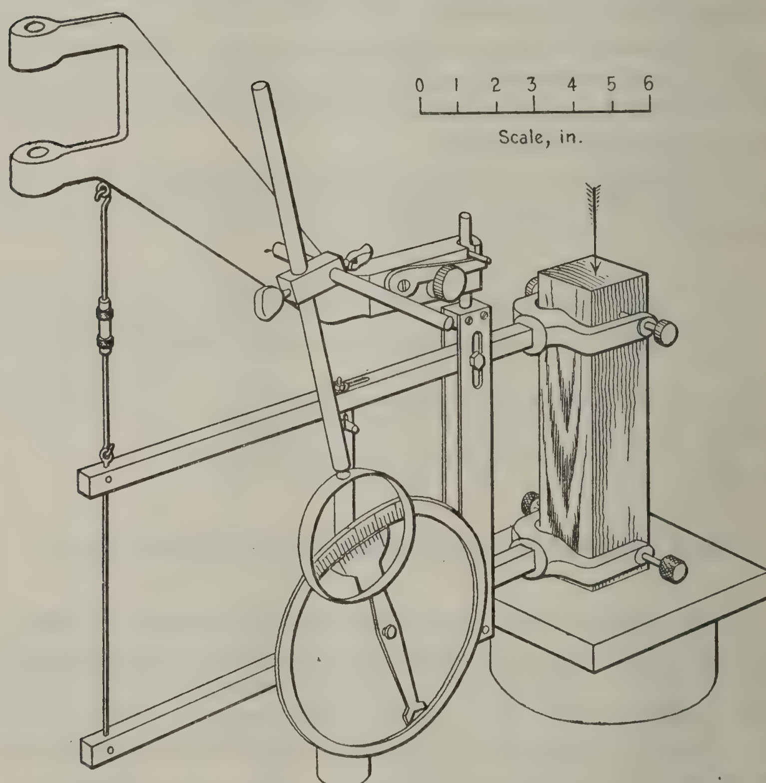

FIG. 9.—Diagrammatic Sketch of U. S. Forest Service Compressometer and Method of Conducting Compression-Parallel-to-Grain Test.

Splintering tension (see Fig. 7*c*);
Brash tension (see Fig. 7*d*);
Compression (see Fig. 7*e*);
Horizontal shear (see Fig. 7*f*).

[1] The term "cross grain" shall be considered to include all deviations of grain from the direction of the longitudinal axis or longitudinal edges of the specimen. It should be noted that spiral grain may be present even to a serious extent without being evident from a casual observation.

In case two or more kinds of failures develop, all shall be described in the order of their occurrence; thus, compression followed by splintering tension. The failure shall be sketched on the data sheet.

Weight and Moisture Content.

49. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut near the failure.

(*B*) *Compression Parallel to Grain.*

Size of Specimens.

50. The compression-parallel-to-grain tests shall be made (1) on nominal 2 by 2 by 8-in. specimens, or (2) on specimens of the special form shown in Fig. 8. The actual cross-section dimensions, or the diameter at minimum section, and the length shall be measured.

Advantage of Special Form.

51. The specimen with reduced cross-section (Fig. 8) will usually require less care preparatory to test in order to prevent crushing at the ends than the specimen with uniform cross-section throughout the length.

End Surfaces Parallel.

52. Care shall be used in preparing the compression-parallel-to-grain test specimens to make the end surfaces parallel.

Spherical Bearing.

53. A spherical bearing block shall be used and so adjusted for each test as to obtain a uniform distribution of load over the ends of the specimen.

Rate of Application of Load.

54. The load shall be applied continuously throughout the test at a rate of 0.024 in. per minute.

Load-Compression Curves.

55. (*a*) Load-compression curves shall be taken for a 6-in. central gage length on 20 per cent of the specimens. Load-compression readings shall be continued until the elastic limit is well passed, as indicated by the curve.[1] On the other 80 per cent of the specimens the maximum load only will be obtained.

(*b*) Deformations shall be read to 0.0005 in.

(*c*) Fig. 9 illustrates a type of compressometer, using a Johnson dial, which has been found satisfactory for wood testing.

Position of Test Failures.

56. In order to obtain satisfactory and uniform results, it is necessary that the failures be made to develop in the body of the specimen. With specimens of uniform cross-section, this result can best be obtained when the ends are at a very slightly lower moisture content than the body. With green material it will usually suffice to close-pile the specimens, cover the body with a damp or wet cloth and expose the ends for a short time. For air-dry material, it may sometimes be advisable to pile the specimens in a similar manner and place them in a desiccator should the failures in test indicate that a slight end drying is necessary.

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[1] See Fig. 4 of the Appendix for a sample compression-parallel-to-grain data sheet form. Fig. 3 of the Appendix shows a sample computation data card.

Description of Failures.

57. Compression failures shall be classified and described according to their appearance, as follows:

(*a*) *Crushing.*—This term shall be used when the plane of rupture is approximately horizontal. See Fig. 7(*g*).

(*b*) *Wedge Split.*—The direction of the split, that is whether radial or tangential, should be noted. See Fig. 7(*h*).

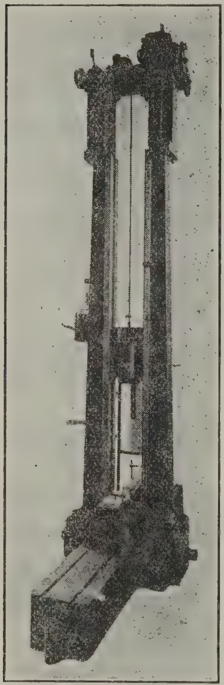

FIG. 10.—Hatt-Turner Impact Machine, Illustrating Method of Conducting Impact Bending Test.

(*c*) *Shearing.*—This term shall be used when the plane of rupture makes an acute angle with the axis of the specimen. See Fig. 7 (*i*).

(*d*) *Splitting.*—See Fig. 7(*j*).

(*e*) *Compression and Shearing Parallel to Grain.*—This failure usually occurs in cross-grained pieces, and should be culled as a compression test. See Fig. 7(*k*). The failure shall be sketched on the data sheet. In addition the location of the failure shall be recorded along with its description.

Weight and Moisture Content.

58. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut from the body near the failure.

Ring and Summer Wood Measurement.

59. After the moisture section has been weighed, the number of rings per inch and the proportion of summer wood shall be measured over a representative inch of cross-section. In determining the proportion of summer wood, it is essential that the end surface be prepared so as to permit accurate summer wood measurement. When the fibers are broomed over at the ends from sawing, a light sanding, planing, or similar treatment of the ends is recommended.

(*C*) *Impact Bending.*

Size of Specimens.

60. The impact bending tests shall be made on nominal 2 by 2 by 30-in. specimens. The actual height and width at the center and the length shall be measured.

Loading and Span.

61. Center loading and a span length of 28 in. shall be used.

Bearing Block.

62. A metal tup of curvature corresponding to the bearing block shown in Fig. 6 shall be used in applying the load.

Placement of Growth Rings.

63. The specimen shall be placed so that the load will be applied through the bearing block to the tangential or flat-sawn surface nearest the pith.

64. The tests shall be made by increment drops in a Hatt-Turner impact or similar machine. See Fig. 10. The first drop shall be 1 in., which shall be increased by 1-in. increments until a height of 10 in. is reached. A 2-in. increment shall then be used until complete failure occurs or until a 6-in. deflection is reached. **Method of Test.**

65. A 50-lb. hammer shall be used when, with drops up to the capacity of the machine (about 68 in. for the small Hatt-Turner impact machine), it is practically certain that complete failure or a 6-in. deflection will result for all specimens of a species. For all other cases a 100-lb. hammer shall be used. **Weight of Hammer.**

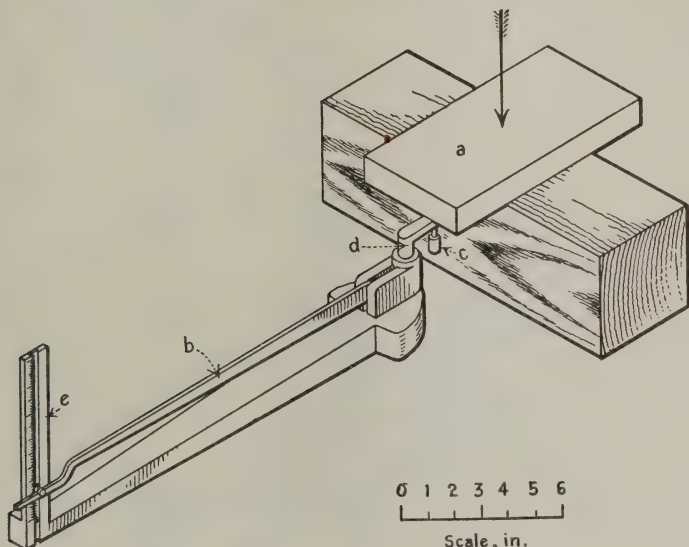

FIG. 11.—Diagrammatic Sketch of Method of Conducting Compression-Perpendicular-to-Grain Test.

66. Graphical drum records,[1] giving the deflection for each drop and the set, if any, shall be made until first failure occurs. This record shall also afford data from which the exact height of drop can be scaled for at least the first four falls. **Deflection Records.**

67. The height of drop causing complete failure, or a 6-in. deflection, shall be observed for each specimen. **Drop Causing Failure.**

68. The failure shall be sketched on the data sheet[2] and described in accordance with the directions for static bending under Section 48. **Description of Failure.**

[1] See Fig. 5 of the Appendix for a sample drum record.

[2] See Fig. 7 of the Appendix for a sample impact bending data sheet form. Fig. 6 of the Appendix shows a sample computation data card.

Weight and Moisture Content. 69. The specimen shall be weighed after the test and a moisture section 1 in. in length shall be cut near the failure.

(D) Compression Perpendicular to Grain.

Size of Specimens. 70. The compression-perpendicular-to-grain tests shall be made on nominal 2 by 2 by 6-in. specimens. The actual height, width, and length shall be measured.

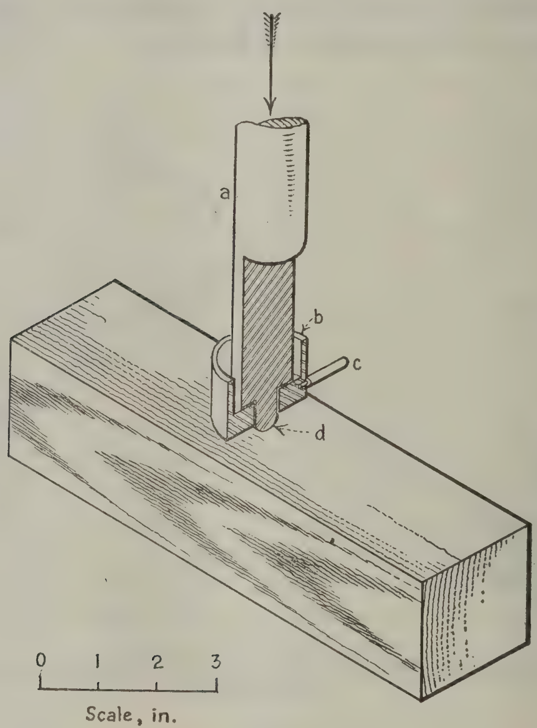

FIG. 12.—Diagrammatic Sketch of Method of Conducting Hardness Test.

Loading. 71. The load shall be applied through a metal bearing plate 2 in. in width, placed across the upper surface of the specimen at equal distances from the ends and at right angles to the length. (See Fig. 11.)

Placement of Growth Rings. 72. The specimen shall be placed so that the load will be applied through the bearing plate to a radial (quarter-sawn) surface.

Rate of Application of Load. 73. The load shall be applied continuously throughout the test at a rate of 0.024 in. per minute.

74. (*a*) Load-compression curves[1] shall be taken for all specimens up to 0.1-in. compression, after which the test will be discontinued. **Load-Compression Curves.**

(*b*) Deflection readings shall be taken to 0.001 in.

75. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut adjacent to the part under load. **Weight and Moisture Content.**

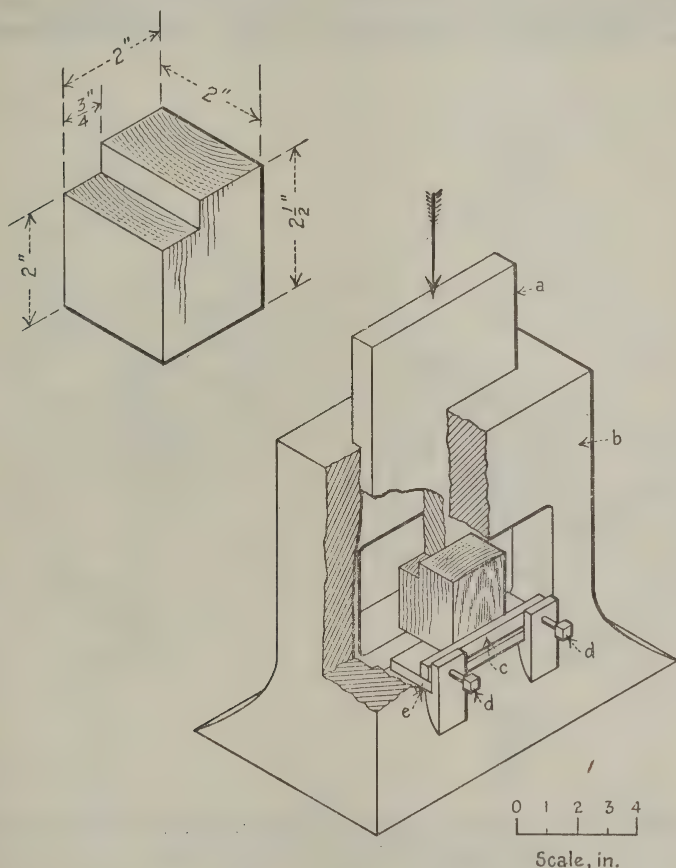

FIG. 13.—Diagrammatic Sketch of Method of Conducting Shear-Parallel-to-Grain Test, with Details of Test Specimen.

(*E*) *Hardness.*

76. The hardness tests shall be made on nominal 2 by 2 by 6-in. specimens. The actual cross-section dimensions and length shall be measured. **Size of Specimens.**

[1] See Fig. 8 of the Appendix for a sample compression-perpendicular-to-grain data sheet form. Fig. 9 of the Appendix shows a sample computation data card.

Method of Test. 77. The modified ball test with a "ball" 0.444 in. in diameter shall be used for determining hardness. (See Fig. 12.) The load at which the "ball" has penetrated to one-half its diameter as indicated by the tightening of the collar against the specimen, shall be observed.

Number of Penetrations. 78. Two penetrations shall be made on a tangential surface, two on a radial surface, and two on each end. The choice between

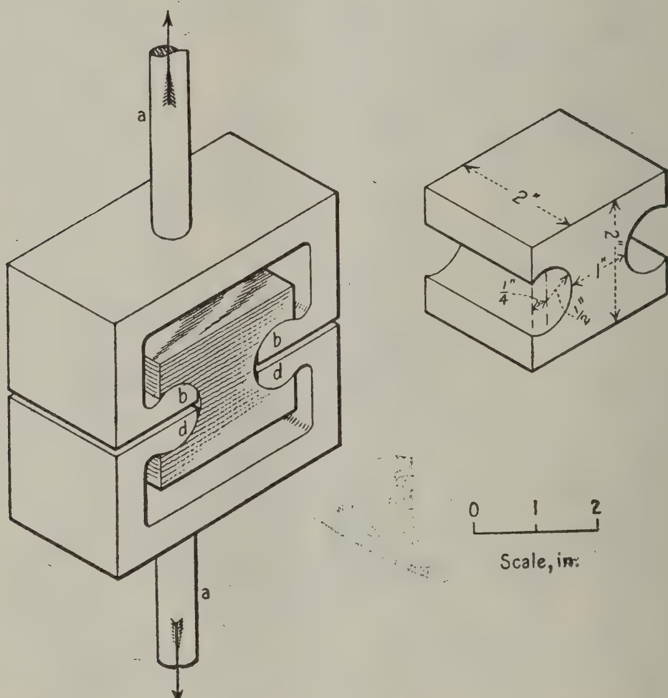

FIG. 14.—Diagrammatic Sketch of Method of Conducting Tension-Perpendicular-to-Grain Test, with Details of Test Specimen.

the two radial and between the two tangential surfaces shall be such as to give a fair average of the piece. The penetrations shall be far enough from the edge to prevent splitting or chipping.[1]

Rate of Application of Load. 79. The load shall be applied continuously throughout the test at a rate of 0.25 in. per minute.

Weight and Moisture Content. 80. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut.

[1] See Fig. 10 of the Appendix for a sample data and computation card.

(*F*) *Shear Parallel to Grain.*

81. The shear-parallel-to-grain tests shall be made on nominal 2 by 2 by $2\frac{1}{2}$-in. specimens notched as illustrated in Fig. 13 to produce failure on a 2 by 2-in. surface. The actual dimensions of the shearing surface shall be measured. **Size of Specimens.**

82. A shear tool similar to that illustrated in Fig. 13, providing a $\frac{1}{8}$-in. offset between the inner edge of the supporting surface and **Method of Test.**

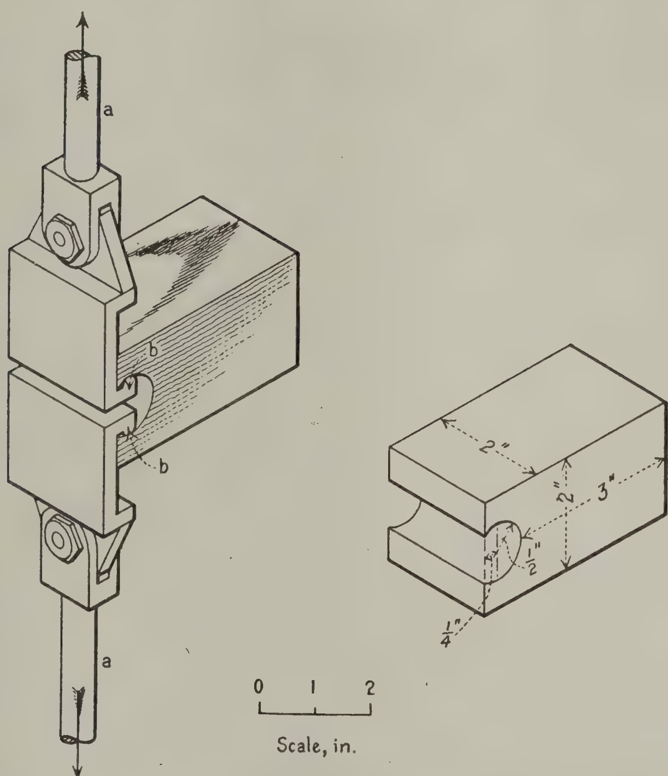

FIG. 15.—Diagrammatic Sketch of Method of Conducting Cleavage Test, with Details of Test Specimen.

the plane along which the failure occurs, shall be used. The load shall be applied and the specimen supported on end-grain surfaces. Care shall be taken in placing the specimen in the shear tool to see that the cross bar is adjusted so that the edges of the specimen are vertical and the end rests evenly on the support over the contact area. The maximum load only will be observed.

Rate of Application of Load.

83. The load shall be applied continuously throughout the test at a rate of 0.015 in. per minute.

Test Failures.

84. The failure shall be sketched on the data card.[1] In all cases where the failure at the base of the specimen extends back onto the supporting surface, the test shall be culled.

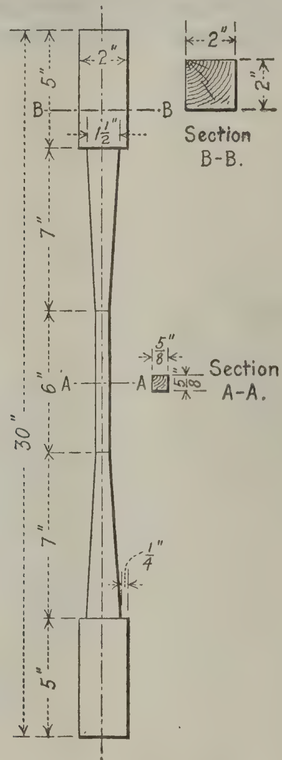

FIG. 16.—Details of Tension-Parallel-to-Grain Test Specimen.

Moisture Content.

85. The portion of the test piece which is sheared off shall be used as a moisture section.

(G) *Tension Perpendicular to Grain.*

Size of Specimens.

86. The tension-perpendicular-to-grain tests shall be made on specimens of the nominal size and shape shown in Fig. 14. The actual width and length at minimum sections shall be measured.

[1] See Fig. 11 of the Appendix for a sample tangential-shear data sheet form.

87. The specimens shall be held during test in grips as shown in Fig. 14. The maximum load only shall be observed. **Method of Test.**

88. The load shall be applied continuously throughout the test at a rate of 0.25 in. per minute. **Rate of Application of Load.**

89. The failure shall be sketched on the data card.[1] **Sketch of Failure.**

90. One of the pieces remaining after failure, or a section split along the surface of failure, shall be used as a moisture specimen. **Moisture Content.**

(*H*) *Cleavage.*

91. The cleavage tests shall be made on specimens of the form and size shown in Fig. 15. The actual width and length at minimum section shall be measured. **Size of Specimens.**

92. The specimens shall be held during test in grips as shown in Fig. 15. The maximum load only shall be observed. **Method of Test.**

93. The load shall be applied continuously throughout the test at a rate of 0.25 in. per minute. **Rate of Operation.**

94. The failure shall be sketched on the data card.[2] **Sketch of Failure.**

95. One of the pieces remaining after failure, or a section split along the surface of failure, shall be used as a moisture specimen. **Moisture Content.**

(*I*) *Tension Parallel to Grain.*

96. The tension-parallel-to-grain tests shall be made on specimens of the size and shape shown in Fig. 16. The actual cross-section dimensions at minimum section shall be measured. **Size of Specimens.**

97. The specimen shall be held during test by plates notched to fit around the test piece and under the 0.25-in. shoulder provided for that purpose. A spherical bearing shall be used on at least one end of the specimen. Maximum load only will be observed. **Method of Test.**

98. The load shall be applied continuously throughout the test at a rate of 0.05 in. per minute. **Rate of Application of Load.**

99. The failure shall be sketched on the data sheet.[3] **Sketch of Failure.**

100. A moisture section about 3 in. in length shall be cut from the minimum section near the failure. **Moisture Content.**

(*J*) *Specific Gravity and Shrinkage in Volume.*

101 The specific gravity and shrinkage in volume tests shall be made on nominal 2 by 2 by 6-in. specimens. The actual cross-section dimensions and length shall be measured. **Size of Specimens.**

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[1] See Fig. 12 of the Appendix for a sample radial-tension-perpendicular-to-grain data sheet form.

[2] See Fig. 13 of the Appendix for a sample radial-cleavage data sheet form.

[3] See Fig. 14 of the Appendix for a sample tension-parallel-to-grain data sheet form.

Determination Method.

102. (*a*) Both specific gravity and shrinkage-in-volume deternations shall be obtained on the same specimen.

(*b*) A carbon impression of the end of the green specimen shall be made on the back of the data card.[1] In like manner, a carbon impression of the same end shall be made after the specimen has been oven dried. (See Paragraph *e*.)

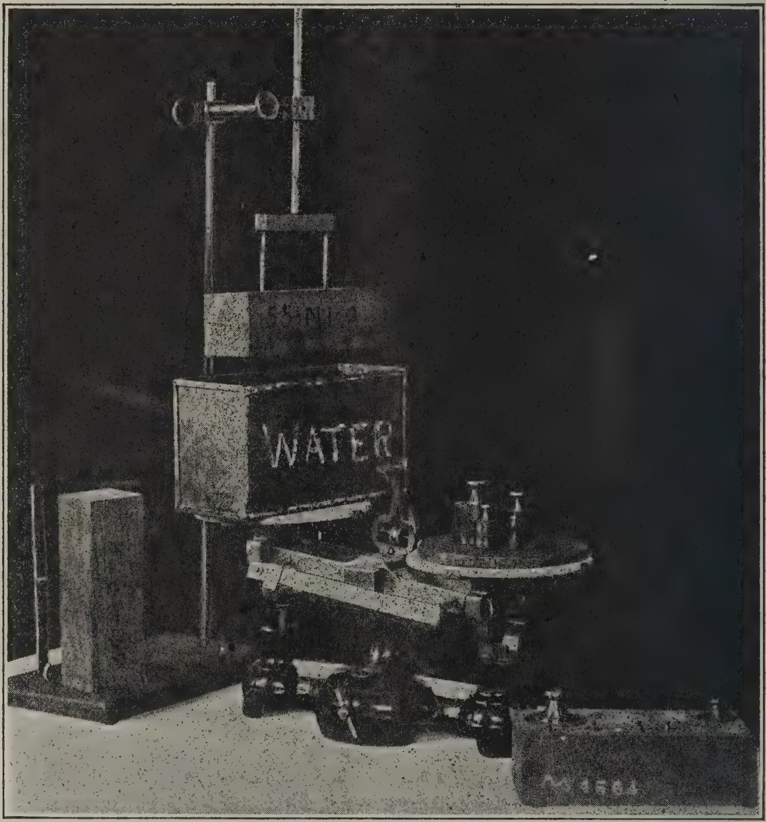


FIG. 17.—Method of Conducting Specific Gravity and Shrinkage-in-Volume Test.

(*c*) The specimen shall be weighed when green and the volume shall be determined by the immersion method.

(*d*) The green specimens after immersion shall be open-piled and allowed to air season under room conditions until approximately constant weight is attained.

[1] See Fig. 15 of the Appendix for a sample specific gravity and shrinkage-in-volume data card form

(e) The specimens shall then be open-piled in an oven and dried at 100° C. until constant weight is reached.

(f) After oven drying, the specimens shall be weighed, and while still warm shall be immersed in a hot paraffin bath, care being taken to remove them quickly to insure a thin coating.

(g) The volume of the paraffin-coated specimen shall be determined by immersion as before.

(h) Fig. 17 illustrates the apparatus used in determining the specific gravity and shrinkage in volume.

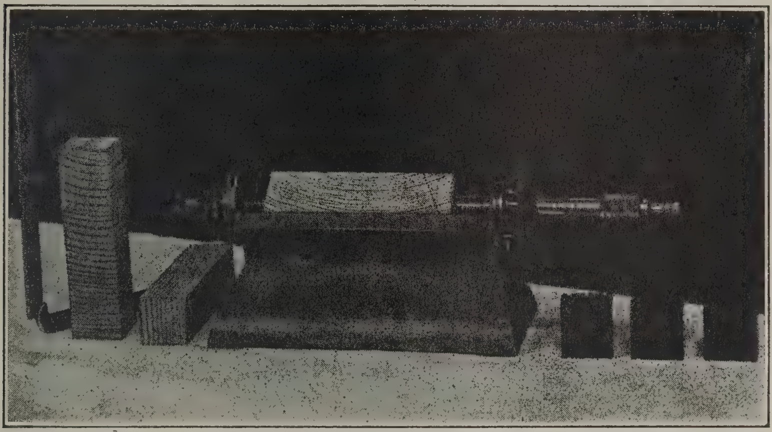


FIG. 18.—Method of Measuring Radial and Tangential Shrinkage.

(K) *Radial and Tangential Shrinkage.*

103. The radial and tangential-shrinkage determinations shall be made on nominal 1 by 4 by 1-in. specimens. The specimen shall be measured across the (4-in.) dimension in which the shrinkage is to be determined. **Size of Specimen.**

104. The specimen shall be weighed when green and after subsequent oven drying. **Weight.**

105. (a) The green specimens shall be open-piled and allowed to air season under room conditions until approximately constant weight is attained. **Drying.**

(b) The specimens shall then be open-piled in an oven and dried at 100° C. until constant weight is attained.

106. Final measurement shall be made on the oven-dry specimen.[1] **Final Measurement.**

[1] See Fig. 16 of the Appendix for a sample radial and tangential-shrinkage data card form.

107. Fig. 18 illustrates the method of making the radial and tangential-shrinkage measurements. An ordinary micrometer of required accuracy is suitable for this work.

(*L*) *Moisture Determination.*

Selection. 108. The sample for moisture determinations of each test specimen shall be selected as heretofore described for each test.

Weighing. 109. Immediately after obtaining the moisture sample, all loose splinters shall be removed and the sample shall be weighed.

Drying. 110. The moisture samples shall be open-piled in an oven and dried at a temperature of 100° C. until constant weight is attained, after which the oven-dry weight shall be determined.

Moisture Content. 111. The loss in weight expressed in percentage of the oven-dry weight as above determined shall be considered the moisture content of the specimen.

VI. TOLERANCES.

(*A*) *Weights.*

Weights. 112. The weight of test specimens and of moisture samples shall be determined to an accuracy of not less than 0.2 per cent.

(*B*) *Measurements.*

Measurements. 113. Measurements of test specimens will be made to the nearest 0.01 in. except in the case of tension parallel to grain, radial shrinkage, and tangential shrinkage, when measurements will be taken to the nearest 0.001 in.

(*C*) *Testing Machine Speeds.*

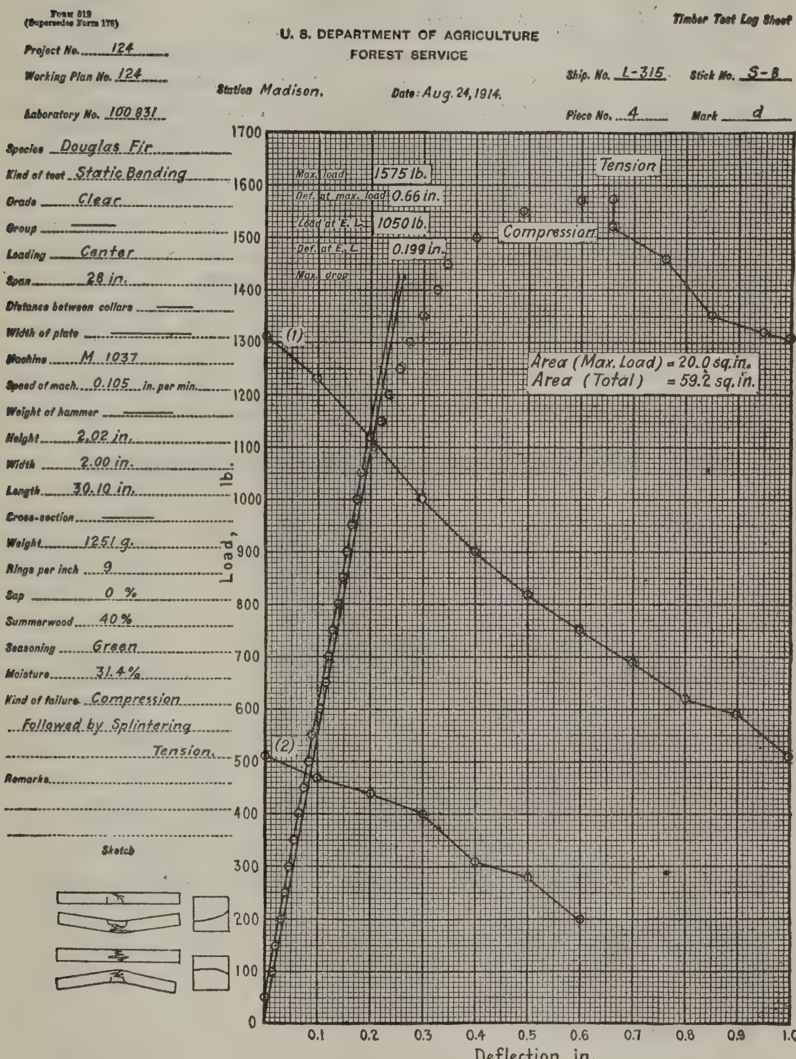
Testing Machine Speeds. 114. In no case shall the testing machine speed used vary more than 25 per cent from that specified for a given test. The load shall be applied continuously, without interruption, at the required speed throughout the test.

VII. CALIBRATION.

Calibration. 115. All apparatus used in obtaining data shall be calibrated at sufficiently frequent intervals to insure accuracy.[1]

[1] The Society has adopted Standard Methods of Verification of Testing Machines (Serial Designation: E 4), 1924 Book of A.S.T.M. Standards.

APPENDIX.



Form 512
(Supersedes Form 176)

U. S. DEPARTMENT OF AGRICULTURE
FOREST SERVICE

Timber Test Log Sheet

Project No. 124
Working Plan No. 124
Station Madison.
Date: Aug. 24, 1914.
Ship. No. L-315
Stick No. S-8
Laboratory No. 100 831
Piece No. 4
Mark d

Species Douglas Fir
Kind of test Static Bending
Grade Clear
Group ———
Loading Center
Span 28 in.
Distance between collars ———
Width of plate ———
Machine M 1037
Speed of mach. 0.105 in. per min.
Weight of hammer ———
Height 2.02 in.
Width 2.00 in.
Length 30.10 in.
Cross-section ———
Weight 1251 g.
Rings per inch 9
Sap 0 %
Summerwood 40 %
Seasoning Green
Moisture 31.4 %
Kind of failure Compression Followed by Splintering Tension.
Remarks

Sketch

FIG. 1.—Sample Data Sheet for Static Bending Test.

STATIC BENDING

L-315 (Ship No.) S-8 (Stick No.) Center Loading 100 831 (Lab. No.)

4 (Piece No.) d (Mark) Station MADISON Date Aug. 24 -'14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 9 Sap 0 % Summerwood 40 % Moisture 31.4 %

Span 28 in. Length 30.10 in. Height 2.02 in. Width 2.00 in. Weight 1251 g.

Specific Gravity		F. S. at E. L.	M. of R.	M. of E.	Shear.	Work to E. L.	Work to Max. Load.	Total Work.	
As Test.	Ov. Dry.								
0.628	0.478	5410	8120	1756	292	0.92	7.1	20.9	

Rings: Up. ⅓ .... Mid. ⅓ .... Low. ⅓ ....

Sum. wood: Up. ⅓ .... Mid. ⅓ .... Low. ⅓ ....

Defects ....

Failure Compression Followed by Splintering Tension

S—1434

MOISTURE DISTRIBUTION.

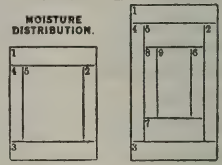


Fig. 2.—Sample Computation Data Card for Static Bending Test.

COMPRESSION PARALLEL TO GRAIN

L-315 (Ship. No.) E-11 (Stick No.) 101 101 (Lab. No.)

1 (Piece No.) d-1 (Mark) Station Madison Date Aug. 25, 1914 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 9 Sap 90 % Summerwood 38 % Moisture 57.6 %

Length 8.00 in. Cross section 2.01 in. X 2.01 in. Weight 365 g.

Specific Gravity		Max. Load	Crush. St. at E. L.	Max. Crush. St.	M. of E.	Load at E.L.	Def. at E.L.
At Test	Ov. Dry						
0.690	0.438	14 760	3220	3660	1992	13 000	0.0097

Defects ....

Failure Shear at Top

S—1468

Fig. 3.—Sample Computation Data Card for Compression-Parallel-to-Grain Test.

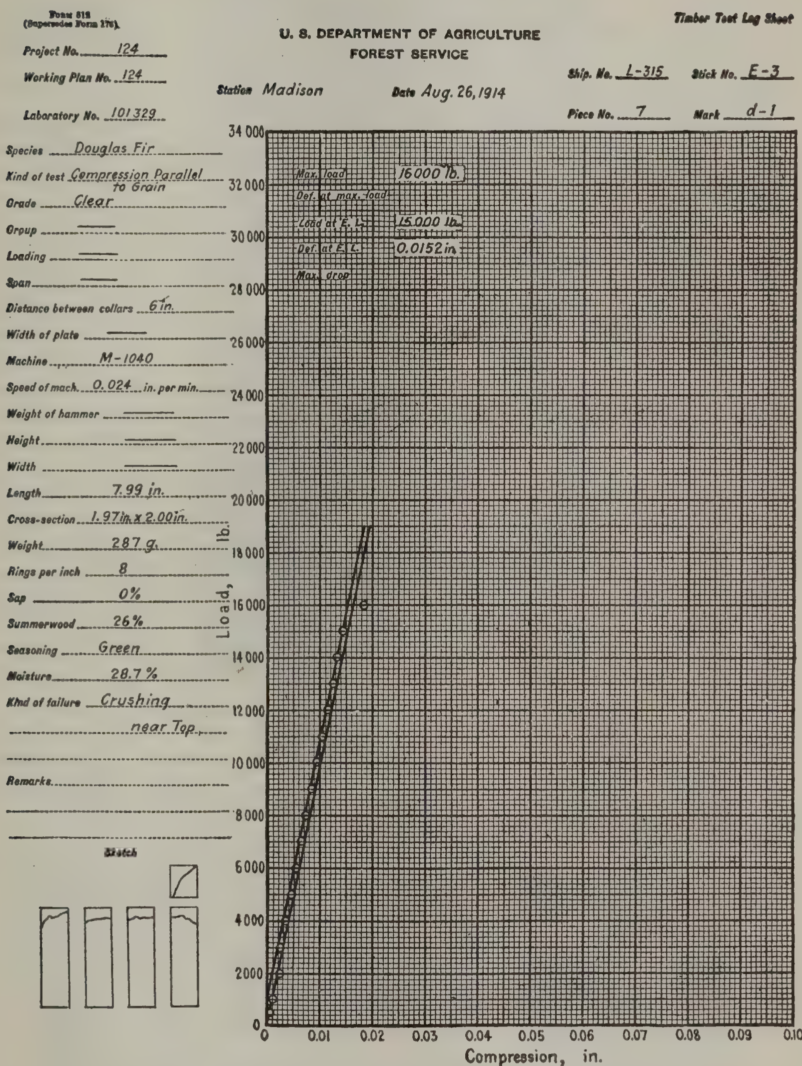


FIG. 4.—Sample Data Sheet for Compression-Parallel-to-Grain Test.

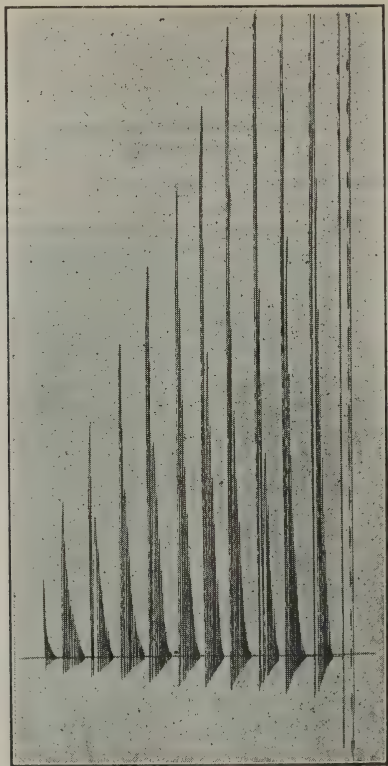


FIG. 5.—Sample Drum Record of Impact Test.

L-315 (Ship. No.) E-12 (Stick No.) **IMPACT BENDING** 101151 (Lab. No.)

1 (Piece No.) C (Mark) Station MADISON Date Aug. 20, 1914 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap 100 % Summerwood 30 % Moisture 61.4 %

Hammer 50 lbs. Span 28 in. Length 29.94 in. Height 2.00 in. Width 2.00 in. Weight 1370 g.

Drop No.	Head.	Def.	(Def.+d)².	Set.	Drop No.	Head.	Def.	(Def.+d)².	Set.		
1	1.0	0.13	0.017		11	12.0	0.50	0.250		Sp. Gr. (at test),	0.698
2	2.0	0.18	0.032		12	14.0	0.55	0.302		Sp. Gr. (oven dry),	0.432
3	3.0	0.22	0.048		13	16.0	0.62	0.384		F. S. at E. L.,	10 610
4	4.0	0.26	0.068		14	18.0	0.67	0.593		M. of E.,	1776
5	5.0	0.30	0.090		15					E. Resil.,	3.51
6	6.0	0.34	0.116		16					Max. Drop,	22 in.
7	7.0	0.36	0.130		17					d,	0.010
8	8.0	0.38	0.144		18					H	7.88
9	9.0	0.43	0.185		19						
10	10.0	0.46	0.212		20					Δ	0.39

Failure: Compression Followed by Splintering Tension.

FIG. 6.—Sample Computation Data Card for Impact-Bending Test.

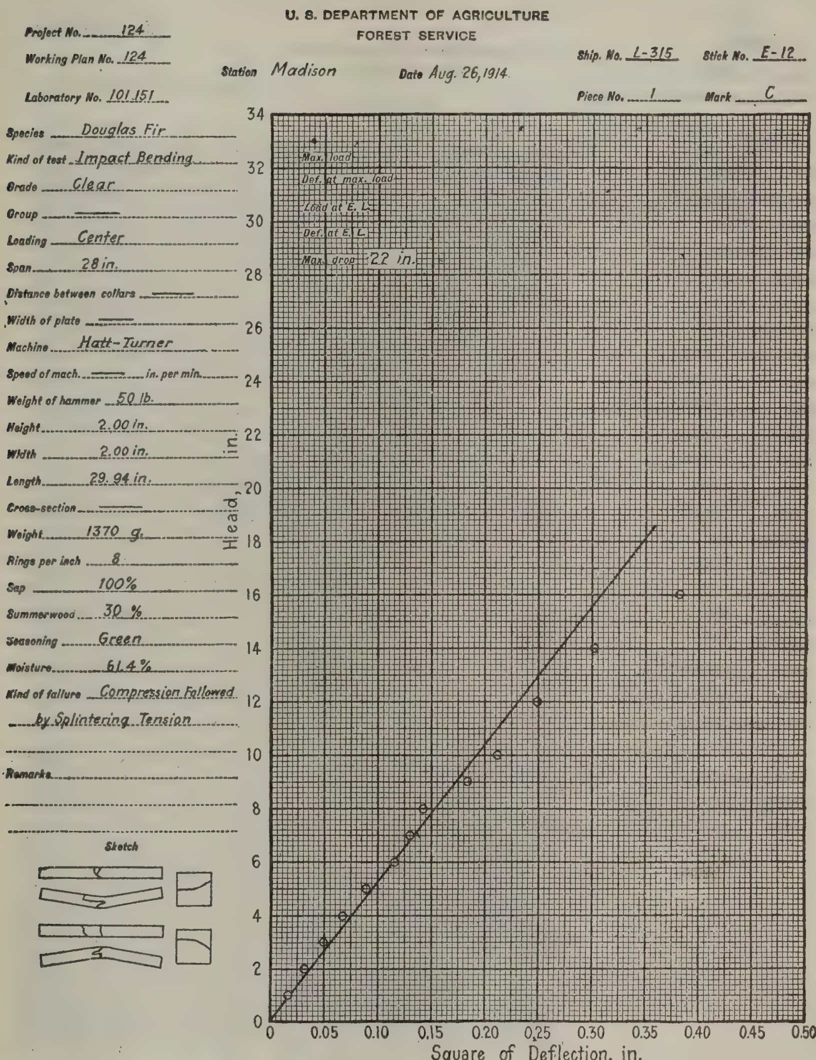


FIG. 7.—Sample Data Sheet for Impact-Bending Test.

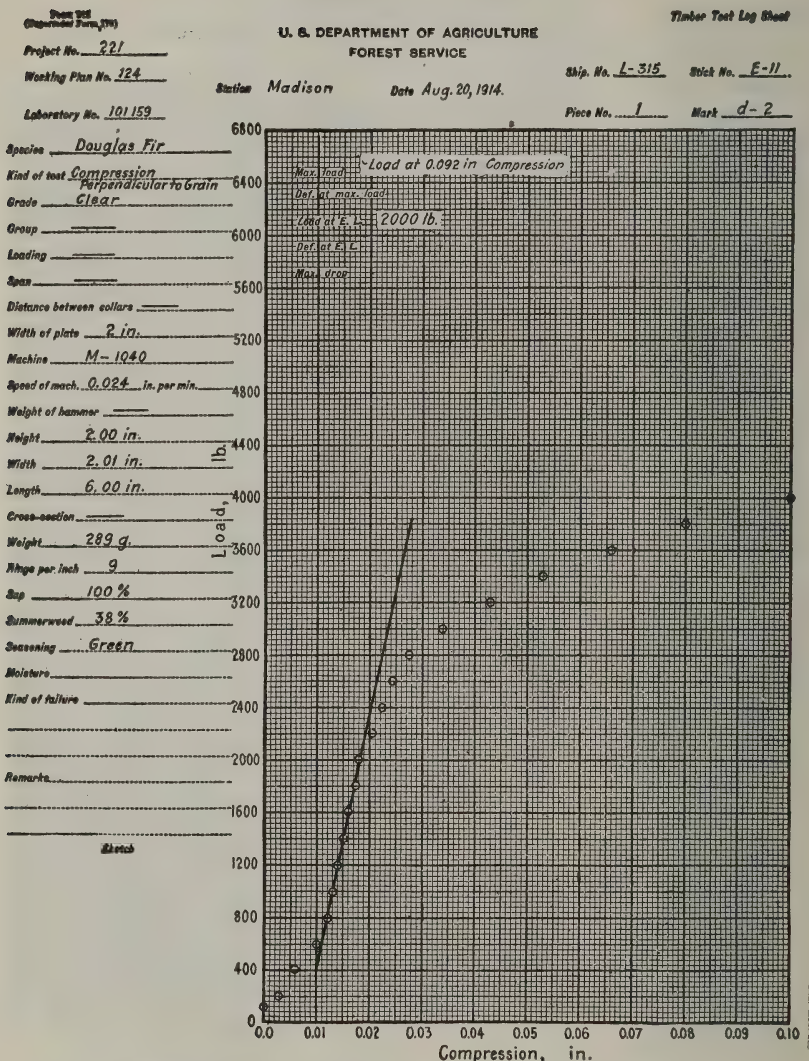


FIG. 8.—Sample Data Sheet for Compression-Perpendicular-to-Grain Test.

L-315 (Ship. No.) E-11 (Stick No.) COMPRESSION PERPENDICULAR TO GRAIN 101159 (Lab. No.)

1 (Piece No.) d-2 (Mark) Station MADISON Date Aug. 20, 1914 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 9 Sap 100 % Summerwood 38 % Moisture 62.1 %

Width of plate 2 in. Length 6.00 in. Height 2.00 in. Width 2.01 in. Weight 289 g.

Specific Gravity		Load at E. L.	Crush. St. at E. L.	Δ ÷ h	
At Test.	Ov. Dry				
0.732	0.452	2000	498		

8—1464

FIG. 9.—Sample Computation Data Card for Compression-Perpendicular-to-Grain Test.

HARDNESS

L-315 (Ship. No.) E-10 (Stick No.) 101170 (Lab. No.)

1 (Piece No.) d-4 (Mark.) Station MADISON Date Aug. 20-14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap — % Summerwood 33 % Moisture 31.7 %

Length 6.01 in. Cross section 2.00 in. x 2.00 in. Weight 246 g.

	Specific Gravity		Radial Surface.	Tangential Surface.	End Surface.
	At Test.	Ov. Dry.			
1	0.622	0.472	460	570	525
2			520	460	500
3					510
4					510
Avg.,			490	515	511
Avg. Rad. and Tang.,			502		

SKETCH.

8—1432

FIG. 10.—Sample Data and Computation Card for Hardness Test.

Tangential SHEAR

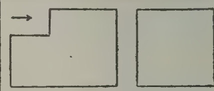
L-315 (Ship. No.) E-12 (Stick No.) 101 183 (Lab. No.)

1 (Piece No.) C-5 (Mark) Station MADISON Date Aug. 21-14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap 75 % Summerwood 30 % Moisture 80.0 %

Shearing Area	Max. Load	Shearing Str.	Time		Sketch
2.02 x 2.01	3600	887			



8—1465

Fig. 11.—Sample Data and Computation Card for Tangential-Shear-Parallel-to-Grain Test. (Surface of failure tangential.)

R TENSION PERPENDICULAR TO GRAIN

L-315 (Ship. No.) E-12 (Stick No.) 101 189 (Lab. No.)

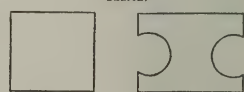
1 (Piece No.) C-6 (Mark) Station MADISON Date Aug. 21-14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap 80 % Summerwood 30 % Moisture 44.2 %

Height.	Width.	Length.	Maximum Load.	Tension. Lbs. per sq. in.
	2.01	0.97	533	273

Sketch.



8—2515

Fig. 12.—Sample Data and Computation Card for Radial-Tension-Perpendicular-to-Grain Test. (Surface of failure radial.)

R CLEAVAGE

1-315 (Ship. No.) E-12 (Stick No.) 101 177 (Lab. No.)

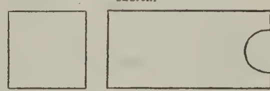
1 (Piece No.) C-3 (Mark) Station MADISON Date Aug. 21, -14 124 (Project No.)

Species Douglas Fir *Grade* Clear *Seasoning* Green

Rings 8 *Sap* 75 % *Summerwood* 30 % *Moisture* 31.6 %

HEIGHT.	WIDTH.	LENGTH.	MAX. LOAD.	LOAD PER INCH WIDTH.
	2.01	2.98	365	182

SKETCH.



8—1433

FIG. 13.—Sample Data and Computation Card for Radial-Cleavage Test. (Surface of failure radial.)

TENSION PARALLEL TO GRAIN

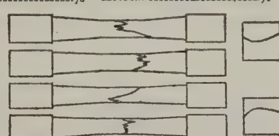
606 (Ship. No.) HM-14 (Stick No.) 347 416 (Lab. No.)

2 (Piece No.) 5-f (Mark) Station MADISON Date Jan. 23, '19 221-1 (Project No.)

Species Douglas Fir *Grade* Clear *Seasoning* Green

Rings 10 *Sap* — % *Summerwood* 31 % *Moisture* 32.5 %

CROSS SECTION.	LENGTH.	MAXIMUM LOAD.	TENSION. Lbs. per sq. in.
0.641 x 0.617		4670	11 810



8—2515

Fig. 14.—Sample Data and Computation Card for Tension-Parallel-to-Grain Test.

SPECIFIC GRAVITY AND VOLUMETRIC SHRINKAGE

L-315 (SHIP. NO.) S-6 (STICK NO.) 101197 (LAB. NO.)

1 (PIECE NO.) d-8 (MARK) STATION. Madison, Aug. 20,-14 (DATE) 124 (PROJECT NO.)

SPECIES Douglas Fir

NOMINAL SIZE OF SPECIMEN 2 in. x 2 in. x 2 in. % SAP 0 % SUMMER WOOD 40

	DATE	RINGS PER INCH	WEIGHT, GRAMS	% MOIST	VOLUME C. C.	SPECIFIC GRAVITY	WEIGHT, LBS. PER CU. FT.	X % VOL. SHRINKAGE
GREEN	Aug. 20,-14	8	253	33.2	398	0.636	39.6	13.8
AIR DRY								
KILN DRY								
OVEN DRY	Sep. 25,-14		190		343	0.554	34.5	

X BASED ON ORIGINAL VOLUME (GREEN, AIR DRY, KILN DRY)

NOTE—USE BACK OF CARD FOR CARBON IMPRESSIONS

1ST WT.

2D WT.

REMARKS: VOL.

3—3034

Fig. 15.—Sample Data and Computation Card for Specific Gravity and Shrinkage-in-Volume Test.

SHRINKAGE—RADIAL AND TANGENTIAL

L-315 (SHIP. NO.) (STICK NO) 101200 101199 (LAB. NOS.)

1 (PIECE NO.) d (MARK) STATION—MADISON, WISCONSIN 124 (PROJECT NO)

SPECIES Douglas Fir

NOMINAL SIZE OF SPECIMEN 1 in. x 4 in. x 1 in.

SEASONING	DATE	RINGS PER INCH	% SAP	% SUMMERWOOD	WIDTH INCHES	WEIGHT GRAMS	% MOISTURE	x % SHRINKAGE
				RADIAL				
GREEN	Aug. 19-14	11	30	41	4.006	49.8	66.5	
OVEN-DRY	Oct. 5-14				3.808	29.9		4.9
				TANGENTIAL				
GREEN	Aug. 19-14	12	95	34	4.016	64.0	119.1	
OVEN-DRY	Oct. 5-14				3.632	29.2		9.5

X BASED ON GREEN WIDTH

Fig. 16.—Sample Data and Computation Card for Radial and Tangential-Shrinkage Tests.

TENTATIVE METHODS OF CONDUCTING STATIC TESTS OF TIMBERS IN STRUCTURAL SIZES[1]

Serial Designation: D 198 - 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924

Although numerous tests of timber in structural sizes have been made for various purposes, there has been a general lack of uniformity of test procedure. Consequently, it has not been possible to correlate data from different sources, nor have the results obtained by different investigators contributed their full measure of usefulness toward an increasing fund of knowledge. Study and experience have shown the extent to which various factors, such as loading appliances, rate of loading, etc., influence test results, and it is with the thought of standardizing these and other factors that this test procedure is prepared. The plan has been to cover very definitely the tests of certain types of timbers such as bridge stringers and joists and at the same time to suggest procedure, so far as possible, for other structural timber forms. Summarized, the underlying purpose is the establishment of practice which will afford uniform results of the greatest ultimate value from all tests of timbers and at the same time permit the correlation of data so far as possible with existing data on timbers of certain species.

It is not the intention to discourage the use of methods other than those herein prescribed whenever the testing problem is of such nature as to warrant their application. However, experience has emphasized the importance of the requirements that have been specified in these methods, and variations in apparently minor details will often seriously affect the data obtained. It is recommended that any variations from the methods herein prescribed shall be recorded.

The field of timber testing is so broad, and involves so many objectives, particularly when built-up or manufactured articles are considered, that it is obviously difficult, if not impossible, to formulate correct procedure for all such cases. Ample opportunity, therefore, still awaits the application of initiative to special testing problems and promotion of progress in the art. It is hoped, however, that the principles incorporated in the proposed methods will at least serve to some extent as a guide in tests of even a special nature.

The test methods presented are the outgrowth of years of experience and research, largely on the part of the United States Forest Service. Such sample data forms are included as were thought helpful to investigators in

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. J. A. Newlin, Secretary of Committee D-7 on Timber, Forest Products Laboratory, Madison, Wis.

systematizing records. No attempt has been made to cover methods of computation or analysis, as these were considered to lie outside the scope of the standard. Data properly recorded can be analyzed at will, but fundamental differences in test procedure present a barrier beyond which analysis and correlation of results cannot proceed with any degree of assurance.

Purpose of Tests.

1. Tests of timbers in structural sizes are made to afford:

(*a*) Data upon which to base the formulation of grading rules and specifications.

(*b*) Data upon which to establish the relation between the strength properties of structural timbers and of small clear specimens cut from them, and for use in the establishment of allowable stresses.

(*c*) Data as to the influence of defects on the mechanical properties of timbers.

(*d*) Data on the strength properties of different species in structural sizes.

(*e*) Data for use in checking existing formulas relating to structural timbers.

(*f*) Data as to the influence of seasoning on the mechanical properties.

(*g*) Data as to the effect of form and shape of specimen on the properties.

(*h*) Data as to the influence of preservatives and methods of preservative treatment on the mechanical properties.

Classes of Tests.

2. In general, tests of timbers are divided into two classes, as follows:

(*a*) Major tests, that is, tests of principal members under investigation.

(*b*) Minor tests, that is, tests of small clear specimens cut from the principal members.

Kind of Tests.

3. (*a*) The principal tests of such structural timbers as bridge stringers or joists are: Static bending, compression parallel to grain, and compression perpendicular to grain.

(*b*) The accompanying minor tests on small clear specimens are: Static bending, compression parallel to grain, compression perpendicular to grain, hardness, and shear parallel to grain.

MATERIAL

I. COLLECTION

(*A*) *Selection*

Authentic Identification.

4. The material for test shall be selected by one qualified to identify the species.

5. In any given case, the number of test specimens shall be determined from due consideration of the purpose in mind, since definite instructions cannot be given to cover all cases. The following procedure applies particularly to full-size timbers, such as bridge stringers and joists, and incidentally to other types, as fabricated beams, to which special reference will be made in small-type paragraphs. **Procedure for Bridge Stringers, Joists, etc.**

6. (*a*) For tests of timbers of any given species in structural sizes, such as bridge stringers, requiring a range in grade of material, at least forty 32-ft. or eighty 16-ft. green unsurfaced timbers should be selected in a manner that will provide representative material. The length of specimens required for test is dependent upon the depth. For suggested lengths of timbers other than bridge stringers, see Section 22. **Number and Size of Major Specimens.**

(*b*) Timbers 8 by 16 in. or 6 by 12 in. in nominal cross-section are especially recommended.

7. Each 32-ft. timber shall be cut in half to furnish one 16-ft. specimen to be tested green and one to be tested air-dry, thus affording end matching. Where only 16-ft. material is available, the timbers shall be selected and matched in pairs, one timber to be tested green and the other to be tested air-dry. In matching these 16-ft. timbers all factors influencing their strength should, so far as practicable, be considered. Some of the most important factors are density, rate of growth, direction of grain (straight, diagonal, or spiral), character and location of defects (such as knots, checks, etc.), position of specimen in tree with respect to cross-section or height, and the characteristics of individual trees. **Selection and Matching of Specimens.**

(*B*) *Field Marking*

8. At the time of selection a mark shall be made on each timber to designate the butt end. **Butt Designation.**

9. All material collected at a given source and shipped at one time shall be given a shipment number or other designation, which shall be incorporated in the test records. **Shipment Number.**

(*C*) *Field Descriptions*

10. Field notes fully describing the material shall be carefully made by the collector. These notes shall, so far as possible, supply data as outlined in Table I and shall be forwarded for incorporation in the record. **Field Descriptions.**

(D) *Preparation for and Records of Shipment*

Preparation for Shipment. 11. (*a*) The ends of all green timbers shall be given a coating of paint or other suitable substance to retard end drying and end checking, care being taken to retain the butt designation. They shall be close-piled and before and during shipment shall be kept covered with damp shavings or other substance to prevent drying. If conditions are particularly favorable to the development of molds, stains, or decay, the specimens shall first be brushed or sprayed with a dilute solution of copper sulfate, or similar toxic solution.

(*b*) Record shall be made of the shipment routing, bill of lading, kind of shipment, date of shipment, and condition of material when shipped.

TABLE I.—SHIPMENT DESCRIPTION FIELD NOTES.

Project No., *XC*	Shipment No., *012*
Tree No., *1*	Species, *Longleaf pine*
Locality cut: State, *Florida*	County, *Nassau*
Township, *T3N: R25E*	Date cut, *April, 1914*
Slope, *Level.* Absolute elevation, *38 ft.*	Aspect, ...
Undergrowth, *Small amount—scattered*	Soil, *Sandy*
Height, *110 ft.* Seedling or sprout, *Seedling*	Age, *241 years*
Crown, *Large, well developed*	D.B.H., *26 in.*
Stem, *Clear, straight*	Date sawed, *May 12, 1914*
How and when transported from woods, *By wagon, May, 1914*	

LIST OF SPECIMENS.

Piece No.	Size of Specimens			Volume, cu. ft.	Feetage, b. m.	Weight, lb. (Show date)
	Length, ft.	Width, in.	Depth, in.			
1	16.10	7.50	15.50	13.00	171	660

REMARKS: Note any other special points which would be of value.

Grouping. (*c*) Record shall also be made of date of receipt of shipment at destination, its condition and the method of storage. The time elapsing between the sawing of green timbers and their testing should be reduced to a minimum.

II. DISPOSITION AT DESTINATION; MARKING AND STORAGE

12. When the material arrives at the testing laboratory, it shall be divided into two matched groups of timbers of approximately like quality as specified in Section 7, one portion to be tested green and the other to be weighed and placed under cover for air seasoning prior to testing. After the 32-ft. timbers have been cut into 16-ft. lengths, the specimens to be air-dried shall be selected alternately from butt and top cuts.

13. The butt end of each 16-ft. specimen shall be designated "*A*" and the top end "*B*". Other symbols shall be given to each timber in classifying it as to density, position in tree, defects, or other factors that are under consideration. The minor test specimens and data forms shall carry such of these symbols as may be necessary to identify them fully. **Marking.**

14. (*a*) The material to be tested green shall be close-piled on skids which are free from contact with the soil, and shall be kept covered. It shall not be held long enough to permit seasoning or damage from checking, decay, or insect attack. **Storage.**

(*b*) The ends of the specimens to be air-dried shall, if necessary, be given a suitable coat of pitch, paint, or other substance to retard checking. The specimens shall be open-piled in a place allowing free access and circulation of air, but shall be protected from sunshine, rain, snow, and moisture from the ground and shall not be subjected to artificial heat.

(*c*) All of the timbers to be air-dried shall be weighed when stored and at sufficiently frequent intervals thereafter to afford accurate data on the progress of seasoning. No material shall be considered thoroughly air-dry and fully conditioned for testing until practically constant weight has been reached.[1] (Wood absorbs and gives off moisture with changing atmospheric conditions; consequently it never comes to absolutely constant weight.)

(*d*) When properly seasoned, the timbers shall be surfaced on four sides before being tested.[2]

TESTS

III. KIND AND NUMBER

(*A*) *Major Tests*[3]

15. (*a*) The structural member proper shall be tested as a beam. (See Sections 19 to 30.) **Major Bending.**

(*b*) A section 60 in. long shall be cut from the "*A*" or butt end of each beam after test, except when end "*A*" fails by shear in the **Compression Perpendicular to Grain.**

[1] The many factors which influence the rate of seasoning, such as species, moisture content, and diverse climatic differences, preclude the establishment of a definite limit of loss in weight which may be permitted in green material prior to test.

[2] A desirable condition for testing air-dry material in small sizes may be considered as about 12-per-cent moisture content. Large timbers, such as bridge stringers, however, usually dry slowly, but may be considered conditioned when, during the season of most rapid drying, successive weighings indicate the moisture content is as low as may reasonably be expected, with due consideration to the climatic conditions of the locality. Such seasoning may require from about one to four years.

[3] The procedure outlined utilizes to best advantage the entire major specimen and if carefully followed will not usually admit the inclusion of specimens which have been seriously strained in the major bending test. The compression specimens referred to in Sections 15(*b*) and 15(*c*) necessarily include such defects as are normal to the material under test.

bending test; in which case the section shall be taken from the "*B*" or top end. A specimen 30 in. long for compression-perpendicular-to-grain test shall be cut from that end of the 60-in. piece which is nearer the point of loading.

Compression Parallel to Grain. (*c*) Two compression-parallel-to-grain specimens, each 6 by 6 by 24 in., shall be taken from the farther end of the 60-in. piece specified in Paragraph (*b*).

(*B*) *Minor Tests*[1]

Material. 16. (*a*) A 50-in. section shall be cut from the end of the beam opposite that required for the 60-in. specimen specified in Section 15 (*b*). Six 2 by 2 by 50-in. sticks shall be taken from this piece. In case defects make it difficult to obtain certain of the other clear specimens hereinafter provided for, they may be secured from the uninjured portion of the minor static bending specimens after the test covered in Paragraph (*b*) below.

Static Bending. (*b*) Six static bending specimens free from defects shall be taken, one from each of the 50-in. sticks specified in Paragraph (*a*).

Compression Parallel to Grain. (*c*) Six compression-parallel-to-grain specimens free from defects shall be taken, one from each of the 50-in. sticks specified in Paragraph (*a*).

Compression Perpendicular to Grain. (*d*) Three compression-perpendicular-to-grain specimens free from defects shall be taken, one from each of three of the 50-in. sticks specified in Paragraph (*a*).

Hardness. (*e*) Three hardness specimens free from defects shall be taken, one from each of three of the 50-in. sticks specified in Paragraph (*a*) other than those from which the specimens specified in Paragraph (*d*) are taken.

Shear Parallel to Grain. (*f*) Twelve shear specimens free from defects shall be cut, two from each of the 50-in. sticks specified in Paragraph (*a*). One specimen from each pair taken from the same stick shall be tested in radial shear (surface of failure radial to growth rings) and the other in tangential shear (surface of failure tangential to growth rings).

IV. GENERAL INSTRUCTIONS

(*A*) *Photographs of Timbers*

Timbers to be Photographed. 17. All air-dried timbers shall be photographed on four sides both before and after test. All green timbers shall be photographed

[1] The procedure outlined utilizes to best advantage the entire major specimen and if carefully followed will not usually admit the inclusion of specimens which have been seriously strained in the major bending test. As specified in Section 26 of the Tentative Methods of Testing Small Clear Specimens of Timber (Serial Designation: D 143 – 24 T) of the American Society for Testing Materials, p. 528, the minors selected shall be free from defects which will affect the properties under consideration.

on four sides after test only. The middle of the length of the beam, the load and support points, and the order of occurrence of the failures shall be indicated so as to show in the photograph; if necessary to bring them out in the photograph the failures should be penciled or pointed.

(*B*) *Moisture Control*

Control of Moisture Content. 18. (*a*) As specified in Section 14 (*c*), the major specimens to be tested in the air-dry condition shall be brought to practically constant weight before test. All tests shall be carried out in such a manner as to prevent large changes in moisture content.

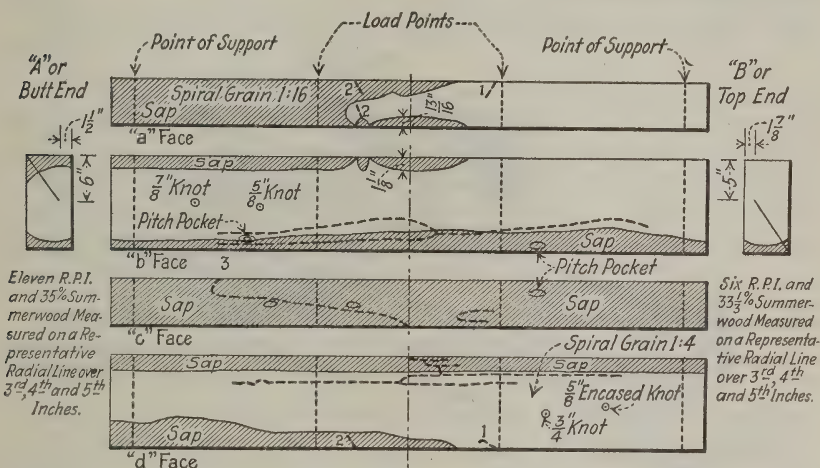


FIG. 1.—Sketch of Structural Size Timber Showing Method of Recording Location and Type of Defects and Other Pertinent Data.

Broken lines indicate test failures, and the accompanying numbers, their order of occurrence.

(*b*) All minor specimens shall be tested immediately after cutting from the majors to prevent change in moisture content.

V. PROCEDURE—MAJOR TESTS

(*A*) *Static Bending Test of Structural Size Timber*

Size and Weight. 19. (*a*) The beam shall be weighed before test. The length and the actual depth and width at the center shall be measured.

(*b*) For non-rectangular beams or beams non-uniform in section data shall be secured from which the exact size and shape of the section at any point throughout the length may be obtained.

Additional Marking. 20. In addition to the markings previously made, the four faces shall be lettered "*a*," "*b*," "*c*," and "*d*," beginning with *a* for the

face to which the load is applied and proceeding successively to the right (clockwise) when viewed from the "*A*" or butt end of the timber.

Sketch of Beam. 21. As part of the record, a sketch shall be made of the faces and ends of each beam showing the size, location, and type of defects, including cross-grain,[1] the distribution of sapwood and heartwood, the location of pith, when present, and the direction of the annual rings (see Fig. 1).[2] Detailed record shall also be made of other defects which may be present, such as crook, bow, cup, and twist.[3] The number of rings per inch and proportion of summerwood at both ends shall also be recorded.

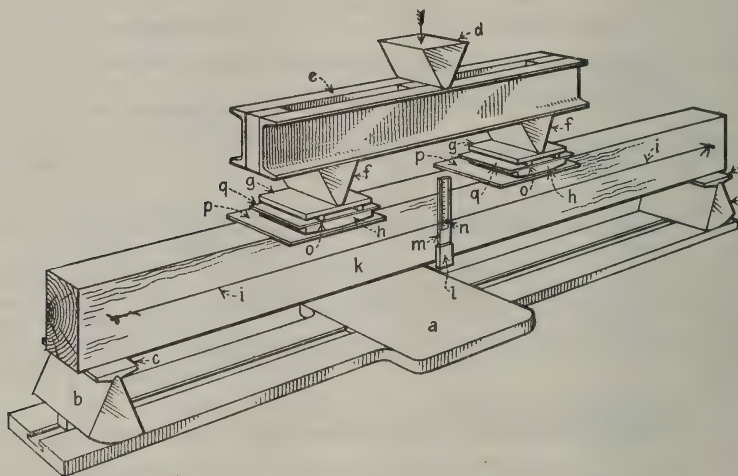


FIG. 2.—Diagrammatic Sketch Showing Method of Conducting Static Bending Test of Structural Sizes of Timber.

Loading, Span and Supports. 22. (*a*) For timbers in structural sizes such as bridge stringers and joists, third-point loading over a span length of 15 ft. shall be used. Knife edges rigidly attached to an auxiliary beam which in turn is hinged by a knife edge shall be used in applying the load. The supporting knife edges shall preferably be of the half-rocker type,

[1] Cross-grained wood is that in which the wood cells or fibers do not run parallel with the axis of the piece. Cross-grain may be classified as spiral, diagonal, dip, wavy, curly, and interlocking. Definitions of these terms may be found in U. S. Department of Agriculture *Circular No. 296*, p. 60. The presence of diagonal and spiral grain is usually indicated by surface checks; these invariably follow the direction of the grain. The amount of cross-grain in any portion of a timber may be expressed by the slope of the fibers (or checks) on each face with respect to the edges of the piece. Thus, a slope of 1 in 10 indicates a departure of the fibers from the direction of the axis of the specimen of 1 in. in 10 in. of length.

[2] Cross-section paper or quadrille-ruled note book paper is recommended for conveniently making this sketch of the beam to scale.

[3] For definitions of these terms, see U. S. Department of Agriculture *Circular No. 296*, p. 65.

and shall be so placed as to rock outward. To prevent accident or damage from their being thrust suddenly outward on failure of the specimen, they should be tied together by means of a slack chain or cable. Metal bearing plates 6 in. in width and not less than $\frac{1}{2}$ in. in thickness and extending entirely across the face of the beam shall be used over the supporting knife edges. (See Fig. 2.)

(*b*) When beams are tested with respect to particular uses, the dimensions shall be such as to simulate actual conditions as closely as practicable.

(*c*) Fabricated beams such as I or box types shall be full size or solid at the loading points without abrupt change from the solid to the normal beam section.

(*d*) Center loading shall not be used for beams over 4 in. in depth when the span-depth ratio is 14 or less.

Bearing Blocks.

23. (*a*) When testing beams under third-point loading on a span equal to 14 times the beam depth the load shall be applied through bearing blocks extending entirely across the face of the beam and having a radius of curvature three times the depth of the beam for a chord length at least equal to the depth of beam. Additional bearing may be obtained by continuing the curvature in both directions at a radius equal to twice the depth of the beam.

(*b*) For beams loaded at the center, the radius of the bearing block shall be one-half that specified for third-point loading under Paragraph (*a*).

(*c*) Whenever possible, span-depth ratios between 11 and 15 shall be used. For span-depth ratios less than 14, the radius of curvature of the bearing blocks shall be proportionally increased over that specified in Paragraphs (*a*) and (*b*).

(*d*) Bearing blocks of the form and size shown in Fig. 3 shall be used at the load points in testing timbers in structural sizes, such as 6 by 12-in. and 8 by 16-in. bridge stringers, on a 15-ft. span.

(*e*) Where the depth of beam is greater than 4 in., a thin steel plate slightly wider than the beam shall be placed between the loading block and the beam. For beams up to 6 in. in depth, the thickness of this plate shall be from $\frac{3}{32}$ to $\frac{1}{8}$ in. For greater depth the thickness of plates shall be increased slightly to about $\frac{5}{32}$ in. for timbers 6 by 12 in. or 8 by 16 in. in cross-section.

(*f*) For fabricated sections such as airplane I and box beams up to 6 in. in depth, the plate shall be from $\frac{1}{16}$ to $\frac{3}{32}$ in. in thickness.

(*g*) Metal bearing plates and rollers shall be used between each bearing block and its corresponding knife edge. The plates between rollers and knife edges shall be not less than $1\frac{1}{4}$ in. in thickness. If the supporting knife edges are of the full-rocker type rather than the

half-rocker type recommended in Section 22 (*a*), rollers shall be placed under one loading knife edge only.

Placement of Specimen in Machine. 24. The beam shall be placed in the testing machine symmetrically with respect to the supports. Parallelism shall be obtained at bearing points. In the case of warped beams, wedges or shims shall be used between the plate and the loading or supporting knife edges or both, as may be necessary to obtain an even bearing before the load is applied.[1] In the case of timbers containing defects, the load shall be applied to the poorer of the loading faces.

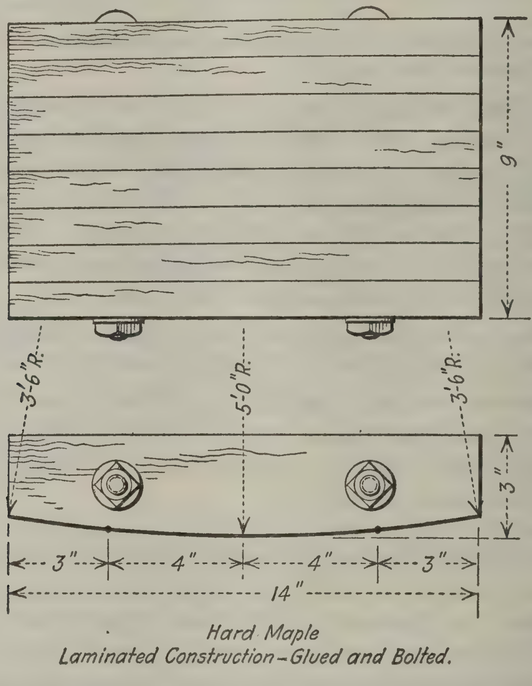


FIG. 3.—Details of Bearing Block for Timbers 12 to 16 in. in Depth When Tested in Third-Point Loading over 15-ft. Span.

Rate of Application of Load. 25. (*a*) The load shall be applied continuously, and with a uniform motion of the movable head throughout the test.

(*b*) *Case 1.*—Beams 4 in. or less in depth when containing defects, or 8 in. or less in depth when free from defects, shall be tested at a rate of fiber strain of 0.0015 in. per inch of outer fiber length per minute.[2]

[1] The use of shims or wedges is recommended in preference to planing because in certain cases the latter procedure may effect changes in section where undesirable.

[2] For tolerance on rate of motion of movable head see Sec. 51.

Case 2.—Beams over 4 in. in depth when containing defects, or over 8 in. in depth when free from defects shall be tested at a rate of fiber strain of 0.0007 in. per inch of outer fiber length per minute.[1]

(*c*) For two symmetrical concentrated loads the rate of motion of the movable head of the testing machine may be calculated from the formula:

$$N = \frac{z\,a}{3\,d}\,(3\,L - 4\,a),$$

where N = the rate of motion of moving head in inches per minute;
z = the unit rate of fiber strain in inches per inch of outer fiber length per minute;
a = the distance from support to adjacent load in inches;
d = the depth of beam in inches;
L = the span in inches.

For example, in testing beams 16 in. in depth under third-point loading and on a 15-ft. span, the rate of motion of movable head shall be 0.26 ±0.065 in. per minute.[1]

(*d*) For center loading, the rate of motion of the movable head of the testing machine may be calculated from the formula:

$$N = \frac{zL^2}{6\,d}$$

where the same legend applies as under Paragraph (*c*).

Load-Deflection Curves.

26. (*a*) Load-deflection curves shall be plotted throughout the test.[2] The load and deflection at first failure, at the maximum load, and at points of sudden change shall be shown on the curve, although they may have to be interpolated because of not occurring at one of the regular load or deflection increments.

(*b*) The test shall be continued until the maximum load has been well passed. In the event of horizontal shear, the maximum load may occur after such failure.

Method of Determining Deflection.

27. (*a*) Deflections of the beam at mid-height at the center of the span with respect to points at mid-height immediately above the supports shall be taken to the nearest 0.01 in.

(*b*) Deflection may be read directly by means of a wire stretched between nails driven at mid-height of the beam immediately above the supports and extending across a scale attached at mid-height at the center of the span. Fig. 2 illustrates the method of test. A

[1] Tolerance in accordance with Section 51.

[2] See Fig. 1 of the Appendix for a sample static-bending data sheet and Fig. 2 of the Appendix for a sample computation data card.

telescope or reading glass will be found useful in observing the deflections. When a reading glass is used parallax shall be prevented by placing a reflecting surface adjacent to the scale in order that the wire and its image may be made to coincide.

(*c*) Where special refinement is desired, deflection readings may be taken simultaneously on both sides of the beam, the wires (Paragraph (*b*)) being supported over free running pulleys and held taut with small suspended weights.

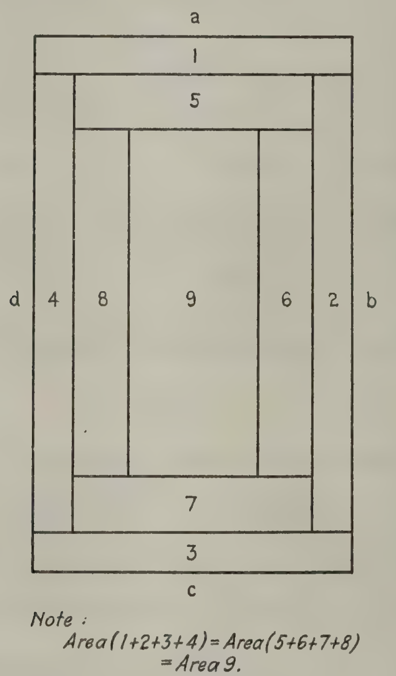


Fig. 4.—Cross-Section of Timber Showing Method of Cutting Up Moisture Distribution Sections.

Description of Failures. 28. The failures shall be described in detail as to type, manner and order of occurrence and position in beam. The descriptions of the failures shall be suitably recorded and correlated with the load-deflection curves. The failures shall be sketched on the drawing of the beam referred to in Section 21, with notations as to the order of their occurrence. The section of the beam containing the failure shall be held for examination and reference until analysis of the data has been completed.

29. A section of the beam at least 2 in. in length shall be retained for purposes of identification and future reference. The number of rings per inch and proportion of summerwood shall be recorded for this section. **Identification Sample.**

30. Two sections 1 in. in length shall be cut from near the place of failure for moisture determinations. One of these shall be used for determining the average moisture content of the test specimen. The other, which may be termed a moisture-distribution section, shall be used for determining the moisture content of different portions of the cross-section and shall be sawn into parts according to the pattern **Specimens for Moisture Determinations.**

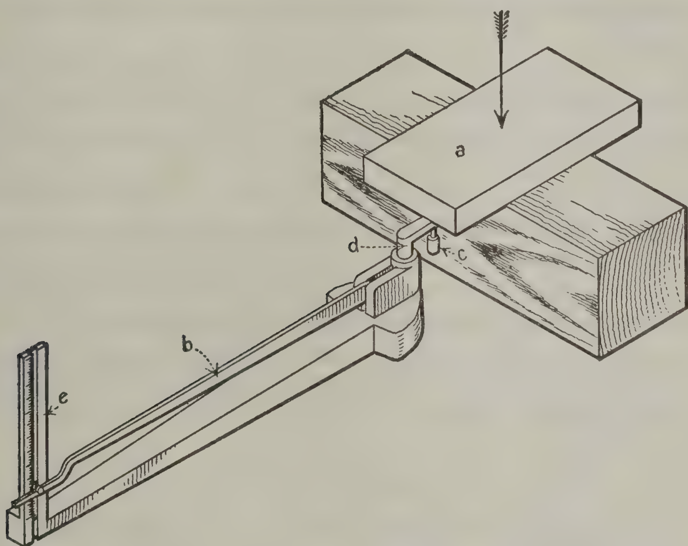


FIG. 5.—Diagrammatic Sketch of Method of Conducting Compression-Perpendicular-to-Grain Test.

shown in Fig. 4. Moisture determinations in each case shall be made in accordance with the procedure specified in Sections 44 to 48.

(*B*) *Compression Perpendicular to Grain*

31. The compression-perpendicular-to-grain tests shall be made on specimens 30 in. in length and of the cross-section of the major beam. The actual width and length shall be measured and recorded, and all defects shall be described. **Size of Specimen.**

32. The load shall be applied through a metal plate 6 in. in width placed across a clear face of the specimen at the center of the length and at right angles to it. The load shall be applied to a face **Loading.**

which corresponds to either the loading or supporting face of the static bending specimen. (Section 24.) (See Fig. 5.)

Rate of Application of Load.

33. (*a*) The load shall be applied continuously throughout the test at a rate of motion of movable head[1] determined from the formula:[2]

$$N = 0.0175\, d^{\frac{4}{9}}$$

where N = the rate of motion of moving head in inches per minute; and

d = the depth of beam in inches.

(*b*) For the specimens 8 by 16 in. in cross-section, for example, the rate of descent of the movable head shall be 0.060 ± 0.015 in. per minute.[3]

Load-Compression Curves.

34. Load-compression curves[4] shall be taken for all specimens until the elastic limit has been well passed, after which the test shall be discontinued. Compression readings shall be taken to 0.001 in.

Weight and Moisture Content.

35. The specimen shall be weighed after test and two sections shall be cut near the place of failure for moisture determinations. One of these shall be used for determining the average moisture content of the specimen. The other, which may be termed a moisture distribution section, shall be used for determining the moisture content of different portions of the cross-section and shall be sawn into parts according to the pattern shown in Fig. 4. Moisture determinations in each case shall be made in accordance with the procedure specified in Sections 44 to 48.

(*C*) *Compression Parallel to Grain*

Size of Specimen.

36. The major compression-parallel-to-grain tests shall be made on the 6 by 6 by 24-in. specimens prepared from the pieces specified in Section 15 (*c*). The actual cross-section dimensions, the rate of growth, and the percentage of summerwood shall be determined and recorded for each specimen. All defects shall be described.

End Surfaces.

37. Care shall be exercised in preparing the compression-parallel-to-grain test specimens to make the end surfaces plane, parallel, and normal to the axis of the specimen.[5]

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[1] For tolerance on rate of motion of movable head see Section 51.

[2] Tests are now in progress to establish more definitely the proper rate of application of load; therefore, the requirement of this section may be changed on the completion of the study.

[3] Tolerance in accordance with Section 51.

[4] See Fig. 4 of the Appendix for a sample compression-perpendicular-to-grain data sheet and Fig. 3 of the Appendix for a sample computation data card.

[5] A sharp fine-toothed saw of either the cross-cut or "novelty" cross-cut type is indispensable for obtaining smooth end surfaces. Power equipment with accurate table guides is especially recommended for this work, though miter-box hand sawing is permissible. Hand planing of the ends, if carefully done, is sometimes advisable.

38. (*a*) A spherical bearing block shall be used to prevent eccentricity of loading. The radius of the sphere shall be as small as practicable, in order to facilitate adjustment of the bearing plate to the specimen. The suspended type of spherical bearing, to be used on top of the specimen, is especially recommended. The size of the compression plate shall be slightly larger than is necessary to encompass the end of the specimen when the latter is centered on the head. **Spherical Bearing.**

(*b*) The adjustment of the suspended spherical bearing block may be accomplished by keeping the compression plate in motion about its bearing as contact is made with the specimen.

(*c*) In the spherical bearing block for use on the weighing table, adjustment shall be made by moving the bearing plate with the specimen centered thereon.

39. The load shall be applied continuously throughout the test at a rate of motion of the movable head of 0.036 ± 0.009 in. per minute.[1] **Rate of Application of Load.**

40. The maximum load obtained shall be recorded, together with a description and sketch of the failure. The section of the specimen containing the failure shall be held for examination and reference until analysis of the data has been completed. **Records.**

41. In order to obtain satisfactory and uniform results, the failures shall be made to develop in the body of the specimen. With specimens of uniform cross-section this may be obtained by having the ends properly prepared and dried to a slightly lower moisture content than the body. With green material, it will usually suffice to close-pile the specimens, cover the body with a damp or wet cloth, and expose the ends for a short time. For air-dry material it may sometimes be advisable, should the failures in test indicate that a slight end drying is necessary, to pile the specimens in a similar manner and place them in a desiccator. **Position of Test Failures.**

42. The specimen shall be weighed after test, and a moisture section shall be cut from the body of the piece near the failure. Moisture determinations shall be made in accordance with the procedure specified in Sections 44 to 48. **Weight and Moisture Content.**

VI. PROCEDURE—MINOR TESTS

43. The procedure for the conducting of minor tests shall conform to the procedure specified in the Tentative Methods of Testing Small Clear Specimens of Timber (Serial Designation: D 143 – 24 T) of the American Society for Testing Materials.[2]

[1] Tolerance in accordance with Section 51.

[2] See p. 528.

VII. MOISTURE DETERMINATIONS

Selection of Samples.

44. The samples for moisture determinations shall be selected as hereinbefore described for each test.

Cutting of "Distribution" Sample.

45. The moisture-distribution specimens shall be cut into sections according to Fig. 4; as specified in Sections 30 and 35.

Weighing of Moisture Sample.

46. Immediately upon obtaining any moisture sample, all loose splinters shall be removed and the sample weighed.

Drying.

47. The moisture samples shall be open-piled in a ventilated oven and dried at a temperature which shall ultimately be increased to 98° C. ± 3° C. until constant weight is attained, after which the resulting weight shall be carefully determined and recorded as the oven-dry weight. For specimens not over 1 in. in length (in the direction of grain) about 48 hours' continuous drying under the conditions specified is sufficient to bring the specimens to constant weight. For drying longer specimens, considerably more time will be required, since the rate of drying from the side grain surface of wood is only about one-sixth that from the end surface.

Moisture Content.

48. The loss in weight expressed as a percentage of the oven-dry weight as above determined shall be considered the moisture content of the specimen.

VIII. TOLERANCES

(*A*) *Weights*

Weights.

49. The weight of test specimens and of moisture samples shall be determined with an error of not more than 0.2 per cent.

(*B*) *Measurements*

Measurements.

50. Measurements of test specimens shall be made to the nearest 0.01 in.

(*C*) *Testing Machine Speeds*

Testing Machine Speeds.

51. In no case shall the testing machine speed used vary more than 25 per cent[1] from that specified for a given test. The load shall be applied continuously at the required speed throughout the test.

IX. CALIBRATION

Calibration.

52. All apparatus used in obtaining data shall be calibrated at sufficiently frequent intervals to insure accuracy.[2]

---

[1] Tests by the U. S. Forest Service show that this speed limitation is necessary in order that the stress shall not vary more than 1 per cent due to variation in rate of fiber strain.

[2] See the Standard Methods of Verification of Testing Machines (Serial Designation: E 4) of the American Society for Testing Materials, 1924 Book of A.S.T.M. Standards.

APPENDIX

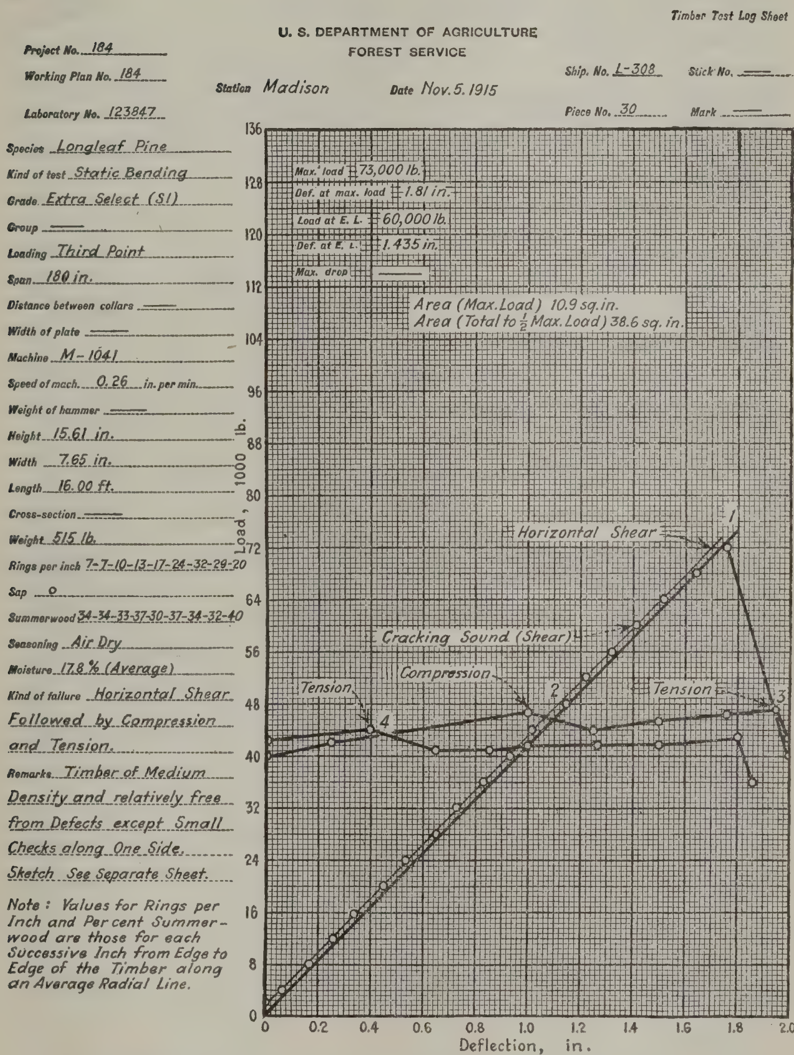


FIG. 1.—Sample Data Sheet for Static Bending Test.

Form 507.
(Revised January, 1918.)

STATIC BENDING

L-308 (Ship No.) —— (Stick No.) One third points Loading 123847 (Lab. No.)

30 (Piece No.) —— (Mark.) Station MADISON, WIS. Date 11-5-15 184 (Project No.)

Species Longleaf Pine *Grade* No. 1 *Seasoning* Air dry

Rings 7-7-10-13-17-24-32-29-20 *Sap* 0 % *Summerwood* 34-34-33 37-30-37-34-32-40 % *Moisture* 17.9 %

Span 180 in. *Length* 16.00 ft. *Height* 15.61 in. *Width* 7.65 in. *Weight* 515½ #

SPECIFIC GRAVITY.		F. S. AT E. L.	M. OF R.	M. OF E.	SHEAR.	WORK TO E. L.	WORK TO MAX. LOAD.	TOTAL WORK.	
AS TEST.	OV. DRY.								
.622	.528	5820	7080	1781	458	1.74	32	11.5	

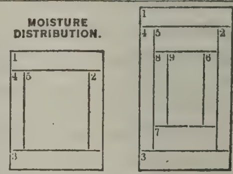
Rings: Up. ¼ —— *Mid.* ½ —— *Low.* ¼ ——

Sum. wood: Up. ¼ —— *Mid.* ½ —— *Low.* ¼ ——

Defects ——

Failure Horizontal Shear followed by Compression and tension.

MOISTURE DISTRIBUTION.



8—1464 19909M

FIG. 2.—Sample Computation Data Card for Static Bending Test.

(Revised January, 1918)

COMPRESSION AT RIGHT ANGLES TO GRAIN

308 (Ship. No.) —— (Stick No.) 124509 (Lab. No.)

30 (Piece No.) —— (Mark) Station MADISON, WIS. Date 11-30-15 184 (Project No.)

Species Longleaf Pine *Grade* 1 *Seasoning* Air dry

Rings 20 *Sap* 0 % *Summerwood* 35 % *Moisture* 17.4 %

Width of plate 5.92 *Length* 30.11 in. *Height* 15.56 in. *Width* 7.51 in. *Weight* 76 #

SPECIFIC GRAVITY		LOAD AT E. L.	CRUSH. ST. AT E. L.	Δ ÷ h	
At Test.	Ov. Dry				
.598	.510	38,000 #	854	——	

8—1464
19908M

FIG. 3.—Sample Computation Data Card for Compression-Perpendicular-to-Grain Test.

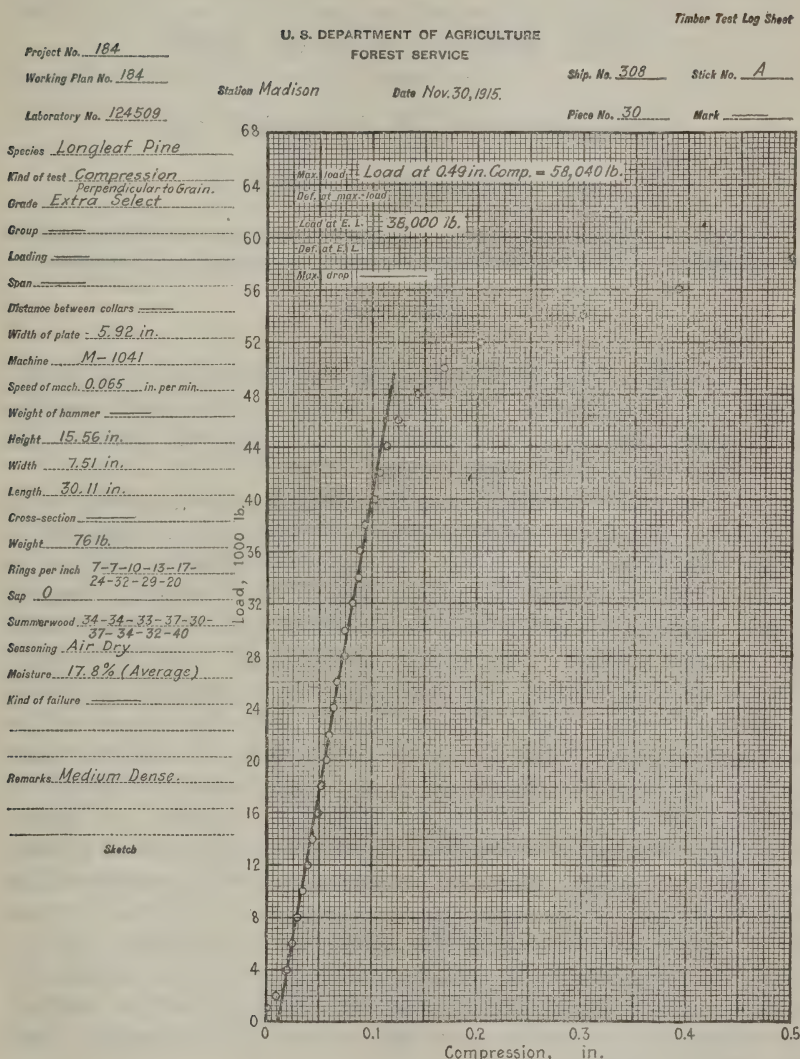


FIG. 4.—Sample Data Sheet for Compression-Perpendicular-to-Grain Test.

TENTATIVE METHOD OF TEST
FOR
COKE RESIDUE OF CREOSOTE OIL[1]

Serial Designation: D 168 - 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923

Apparatus. 1. *Crucible.*—The crucible shall be of platinum and shall have a capacity of 20 to 30 cc. The cover of the crucible shall be of the inverted type, having a depth of about 1 cm., the wall tightly fitting the crucible except for a slight crease.

Procedure. 2. The residue resulting from the distillation test, carried out in accordance with the Standard Methods of Sampling and Analysis of Creosote Oil (Serial Designation: D 38) of the American Society for Testing Materials,[2] shall be poured directly into the tared crucible or into a tin box wherein it may be heated on a water or steam bath, but not over a flame. About 1 g. of the residue shall be weighed into the covered crucible and then placed on a platinum, nichrome, or fire-clay triangle over a Bunsen burner, with the bottom of the crucible 6 to 8 cm. from the top of the burner. The burner flame shall be regulated to a height of 20 cm. while burning free and the crucible shall be exposed to the full flame for 7 minutes. A Meeker burner may be used, or the crucible may be heated for several minutes in an electric furnace. Whatever the method of heating employed, the temperature during the entire 7-minute period shall be not less than 950° C. and should be as near that temperature as possible. At the end of this period the flame shall be removed, the crucible transferred to a desiccator and permitted to cool, after which it shall be weighed. The residue in the crucible after ignition shall be reported as "fixed carbon" (*i. e.*, coke).

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[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. J. A. Newlin, Secretary of Committee D-7 on Timber, Forest Products Laboratory, Madison, Wis.

This tentative method, when adopted as standard, will be added to the Standard Methods of Sampling and Analysis of Creosote Oil (Serial Designation: D 38 - 24), 1924 Book of A.S.T.M. Standards.

[2] 1924 Book of A.S.T.M. Standards.

3. The test shall be conducted in a part of the laboratory free from draughts. **Precautions.**

4. The percentage of coke obtained in accordance with Section 2 shall be calculated on the basis of the original sample of oil. **Calculations.**

Example.—With a retort distillation of 29 per cent of residue at 355° C., the residue containing 28 per cent of fixed carbon:

$$\text{Coke in oil} = \frac{29 \times 28}{100} = 8.1 \text{ per cent}$$

TENTATIVE METHODS OF CHEMICAL ANALYSIS OF ZINC CHLORIDE[1]

Serial Designation: D 199 - 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924.

These methods cover the determination of the percentages of insoluble basic zinc chloride, zinc chloride, and iron present in commercial concentrated solutions of zinc chloride or in fused or granulated zinc chloride, for use in the preservative treatment of wood.

PREPARATION AND STANDARDIZATION OF SOLUTIONS

Standard Ferrocyanide Solution.—Weigh out 43.25 g. of c.p. potassium ferrocyanide and 14 g. of c.p. crystallized sodium sulfite, dissolve in water and make up to 1 liter at room temperature. Shake thoroughly. Standardize against a zinc solution of known concentration prepared from spelter of known zinc content or from c.p. zinc oxide which has been previously ignited. One cubic centimeter of this solution will be equal to approximately 0.01 g. of zinc. The standardization should be carried out as nearly as possible in the same manner as in the estimation of zinc and approximately the same amount of zinc should be present. Keep the solution in a dark bottle. Shake thoroughly before each using and standardize each time it is used.

Uranium Acetate Indicator.—Dissolve 4.4 g. of c.p. uranium acetate, free from sodium, in 100 cc. of hot water and 2 cc. of glacial acetic acid. Use this as an external indicator on a paraffined plate making the drops as nearly 0.05 cc. as possible.

Hydrogen Peroxide.—The usual laboratory reagent is satisfactory, if fresh.

Hydrogen Sulfide Solution.—The usual laboratory reagent is satisfactory.

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. J. A. Newlin, Secretary of Committee D-7 on Timber, Forest Products Laboratory, Madison, Wis.

DETERMINATION OF INSOLUBLE OR BASIC ZINC CHLORIDE

Weigh from 10 to 14 g. of the sample, if fused or granulated, from a stoppered weighing bottle, or place an equivalent quantity, if a solution, into a 600-cc. beaker. Add cold water to 400 cc. Stir the contents of the beaker until solution is complete. Allow to settle over night. Filter the solution through a 12.5-cm. filter paper which has previously been washed, dried, and weighed. Receive filtrate in a 1000-cc. graduated measuring flask. Policeman the beaker, and wash the insoluble matter in the filter paper until the filtrate measures 1 liter. Dry the filter paper containing the insoluble matter over night in an oven heated to 100° C. (212° F.). Cool and weigh between clipped watch glasses. Calculate the increase in weight of filter paper to a percentage of the original sample.[1]

Duplicate determinations should check within 1.3 per cent.

DETERMINATION OF ZINC

(*a*) *Volumetric method.* (For use when manganese chloride does not exceed 0.3 per cent.)

The filtrate obtained from the estimation of insoluble basic zinc chloride and whose volume is exactly 1 liter is shaken and three aliquot portions of 100 cc. each are taken with an accurate 100-cc. pipette and transferred into 450-cc. Griffins beakers. Add to each portion 15 g. of ammonium chloride and 5 cc. of concentrated hydrochloric acid. Dilute to 350 cc. and heat nearly to boiling. Titrate slowly with vigorous stirring using a solution of potassium ferrocyanide as the standard reagent and uranium acetate as an external indicator. The average of the three aliquot portions should be reported.

(*b*) *Volumetric Method.* (For use when the manganese chloride equals or exceeds 0.3 per cent.)

To the aliquot portions taken as described under (*a*), 1 cc. of hydrogen peroxide (2 to 3 per cent) and 10 cc. of ammonia (1:1) shall be added. Stand on steam bath until settled. Filter off the manganese, wash beaker and paper twice with hot water. Dissolve the precipitate in the smallest amount of hydrochloric acid (1:1) in the original beaker, heat until all is dissolved; the volume of the solution should be about 20 cc. Reprecipitate the manganese with 1 cc. of hydrogen peroxide and 10 cc. of ammonia, boil, filter, and wash several times with hot water. Add the filtrate to that obtained in the first separation. Add 15 cc. of concentrated hydrochloric acid

[1] A Gooch crucible may be used in place of the weighed filter paper.

to the combined filtrate and just neutralize with concentrated ammonia, then add 5 cc. of hydrochloric acid in excess. Dilute to 325 cc. and add 25 cc. of saturated hydrogen sulfide water to remove any traces of hydrogen peroxide, heat and titrate as in (*a*).

(*c*) *Gravimetric Method.* (Alternate method, for use when only a limited amount of work is necessary.)

The filtrate obtained from the estimation of insoluble basic zinc chloride and whose volume is exactly 1 liter is shaken and three aliquot portions of 200 cc. each are taken with an accurate pipette and transferred to 450-cc. Griffins beakers. Add to each 4 cc. of concentrated c.p. sulfuric acid. Evaporate on a steam bath; then on a steam plate or hot plate to copious SO_3 fumes, to completely eliminate chlorides. Cool and take up in 100 cc. of hot distilled water. Add 0.5 g. of aluminum powder. Cover with a watch glass. Heat to boiling and boil 5 minutes. Filter through an 11-cm. filter paper. Receive the filtrate in a covered 1000-cc. Griffins beaker. Wash the beaker and filter thoroughly with hot water until a drop of methyl orange indicator placed behind the double fold of filter paper shows no acidity. Exactly neutralize the filtrate with dilute ammonia. Use great care and precision, and carry the neutralization just to the end point.

Add 10 cc. of 0.1 *N* sulfuric acid (3 cc. of concentrated c.p. sulfuric acid in 1000 cc. of water). Dilute to 650 cc. Cover the beaker, and bubble a rapid stream of hydrogen sulfide for one-half to one hour at room temperature.

Settle and filter through a double filter of one 15-cm. and one 11-cm. paper[1] folded together. Transfer the precipitate to the filter paper. Thoroughly policeman the beaker until the zinc sulfide is all removed except a very thin film which clings tenaciously to the glass at the surface of the liquid. Thoroughly wash the beaker and precipitate at room temperature with water saturated with hydrogen sulfide. Repeat washing of filter paper, and precipitate five or six times. Transfer paper and precipitate to an ignited, cooled, desiccated and weighed porcelain crucible of suitable capacity (about 25 to 30 cc.). Carefully dry the paper and precipitate and when dry completely burn off the paper at as low a temperature as possible. When carbon has been completely burned out, ignite the resultant oxide of zinc strongly to as high a temperature as is available, but not higher than can be attained with a laboratory blast lamp with the aid of gas and air. Heat for 30 minutes. After strong ignition, cool the crucible, desiccate and weigh. The increase in weight is zinc oxide. The

[1] Ashless paper should be used.

weight of the zinc oxide multiplied by the factor 1.6749, multiplied by 100, divided by the weight of sample in the aliquot portion taken, equals the percentages of zinc chloride.

The average of the results obtained with the three aliquot portions analyzed shall be reported. Results should agree within 1.5 per cent.

ESTIMATION OF IRON AND ALUMINA

Weigh 10 g. of the sample, if fused or granulated, or place an equivalent quantity, if a solution, into a suitable beaker and dissolve in 100 cc. of water or dilute to 100 cc., if a solution. Add sufficient hydrochloric acid to dissolve any basic zinc chloride. Add a slight excess of bromine water and boil off excess. Neutralize with a weak solution of sodium carbonate until a permanent precipitate of zinc carbonate is obtained. Add three drops of glacial acetic acid and 2 g. of sodium acetate, and boil. Filter and wash. Redissolve the precipitate in the original beaker with hot hydrochloric acid (1:1). Reprecipitate the iron and alumina with a slight excess of ammonia, filter and wash free from chlorine. Ignite in a platinum crucible and weigh as $Fe_2O_3+Al_2O_3$.

TENTATIVE METHOD OF TEST

FOR

WATER ABSORPTION OF SLATE[1]

Serial Designation: D 221 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

It is often desirable to gain some idea of the porosity of a material in question. Actual determinations of the pore space require the use of rather elaborate and refined equipment as well as considerable precision in carrying out the tests. For comparative purposes the absorption test affords a simple and sufficiently accurate means of obtaining the desired information. As applied to slate this test requires somewhat more care and precision than many other materials because of its dense nature and, consequently, the small quantities to be dealt with. Furthermore, the cleavage of slate—that property which permits it to be split into thin sheets of uniform thickness—must be taken into consideration when this test is made. On this account misleading results are often obtained on cubical specimens due to accidental cleavage cracks in the specimens. The shapes of specimens and larger number of specimens recommended in the following procedure are intended to eliminate to a large extent, the inconsistent results which may be obtained on this material.

I. TEST SPECIMENS

Size and Number of Specimens. 1. Six representative specimens of the particular slate under consideration shall be submitted for this test. Each specimen shall be 6 by 6 in. measured along the cleavage planes and ¼ in. in thickness.

Preparation of Specimens. 2. The specimens shall be given a honed finish on all surfaces and edges.

II. PROCEDURE

Drying. 3. The specimens shall be dried in an oven at a temperature between 110 and 120° C. for a period of 24 hours.

Initial Weighing. 4. After drying, the specimens may be cooled in the room for 15 minutes and then weighed. In cases where it is not possible to weigh the specimens immediately upon cooling, they shall be placed in a desiccator. The weights shall be determined to the nearest 0.05 g.

Immersion. 5. After the initial weighing the specimens shall be wholly immersed in water at approximately 20° C. for 48 hours. At the end of this period all faces and edges shall be thoroughly wiped off with

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. D. K. Boyd, Secretary of Committee D-16 on Slate, Structural Service Bureau, Otis Building, Philadelphia.

a dry cloth, and the specimens immediately weighed to the same precision as employed for the initial weights.

6. The percentage of absorption shall be calculated from the formula: **Computation of Results.**

$$\text{Percentage of absorption} = \frac{(B-A)100}{A}$$

where A = dry weight of specimen and B = weight of specimen after immersion.

7. The average absorption of the six specimens shall be reported as the absorption of the slate. If any individual value differs from the average of the six determinations by more than 25 per cent of the average value, the specimen shall be examined for defects, and if the result appears to be due to an imperfect specimen, it shall be discarded. The absorption shall then be reported as the average of the results obtained upon the remaining specimens. The report shall in all cases include the results of all determinations as information. **Reporting Results.**

ALTERNATIVE METHOD

Occasionally it is desirable to obtain results in a shorter period than that required by the above procedure. In such cases, the 48-hour immersion period may be supplanted by an 8-hour boiling period. In this procedure the different steps are the same as the foregoing method up to that of immersion. Instead of allowing the specimens to soak for 48 hours they may be placed in an enameled pan or other suitable vessel, covered with water and boiled for 8 hours. Before the final weighing the specimens shall be cooled by allowing tap water to flow over them for at least 30 minutes. The computation and reporting of results are as outlined in the above procedure. Eight hours boiling has been found to give practically the same saturation as 48 hours soaking.

TENTATIVE METHODS OF FLEXURE TESTING OF SLATE[1]

(DETERMINATION OF MODULUS OF RUPTURE AND MODULUS OF ELASTICITY)

Serial Designation: D 222 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Due to the unique properties of slate the flexure test is better adapted to use for strength and elasticity determinations than either compression or tension tests. Furthermore, several uses of slate are such that these determinations are of special interest and value, besides furnishing comparative data.

The property of slate termed "grain" causes a slab of the material to break transversely in one direction somewhat more readily than at right angles to this direction. For this reason it is desirable to test the strength and elasticity both parallel and perpendicular to the grain.

In the quarrying of slate, blasting is frequently resorted to and for this reason certain portions of the material may have been unduly strained. Low or erratic strength results on some of the test specimens should be regarded in the light of defective material.

A. Modulus of Rupture

I. TEST SPECIMENS

Number and Size of Specimens.

1. Six representative specimens, 12 by $1\frac{1}{2}$ by 1 in. in size, of the particular slate under consideration shall be submitted for this test.

Preparation of Specimens.

2. The slate for test shall have been split to a thickness of approximately $1\frac{1}{4}$ in. and then sawed into strips 12 in. in length by $1\frac{1}{2}$ in. in width. Half of these shall be cut with the length parallel to the grain and the other half with the length perpendicular to the grain. The 12 by $1\frac{1}{2}$ in. faces shall then be planed to smooth surfaces and rubbed down to a thickness of approximately 1 in., care being taken to have the finished faces as nearly parallel as practical.

NOTE.—The shapes of specimens recommended for modulus of rupture and elasticity tests were determined partly by consideration of the proportions best adapted to the respective tests and partly on account of the convenience of preparing them from stock material. The small sizes recommended were first for economy of material and second, to permit the use of lighter testing equipment. Where only the large machines are available it is desirable, in order to obtain consistent results, to use larger specimens. In such cases approximately the same proportionate dimensions should be adhered to for the respective tests.

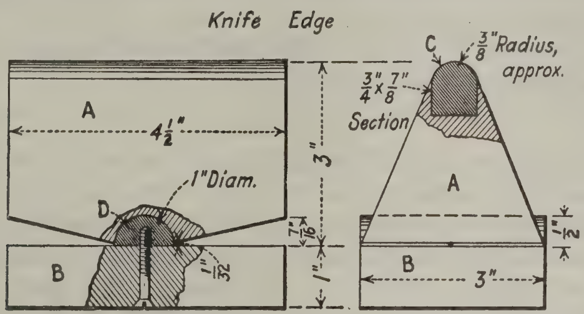
[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. D. K. Boyd, Secretary of Committee D-16 on Slate, Structural Service Bureau, Otis Building, Philadelphia.

II. PROCEDURE

Drying. 3. Before testing, the specimens shall be dried in an oven at a temperature of 110 to 120° C. for 24 hours.

Measuring Specimens. 4. The thickness and the width of the specimen at the middle shall be measured to the nearest 0.01 in.

Testing. 5. The testing machine shall be sensitive to 5 lb. when loaded to 500 lb. The specimen shall be supported flatwise on knife edges of the type shown in Fig. 1, spaced 10 in. on centers. The load shall be



A, B = Cast Iron Wedges.
C, D = Cold-Rolled Steel.

FIG. 1.

applied by a third knife edge at the middle of the span. The rate of loading shall be approximately 100 lb. per minute. The breaking load shall be recorded to the nearest 5 lb.

Computation of Results. 6. The modulus of rupture shall be calculated from the formula:

$$M_f = \frac{3\ wl}{2\ bd^2}$$

where w = breaking load in pounds, l = length of span in inches, b = width of specimen in inches, and d = thickness of specimen in inches.

Reporting Results. 7. The average value of the three specimens cut parallel to the grain shall be reported as the modulus of rupture parallel to the grain, and the average value of the other three, as the modulus of rupture perpendicular to the grain. In case any specimen shows a value 25 per cent lower than the average of its group, it shall be examined for defects and if the low value appears to be due to an imperfection, the average of the other two shall be reported as the modulus of rupture of the group. All determinations shall be reported as information.

NOTE.—The following method of marking the specimens before testing is recommended:

By means of a tri-square the middle section and two end sections, 5 in. distant from the center, may be circumscribed. These marks serve as a guide to the operator in properly centering and aligning the specimen in the testing machine.

B. Modulus of Elasticity

III. TEST SPECIMENS

Number and Size of Specimens. 8. Six specimens, 12 by 4 by $\frac{3}{8}$ in. in size, shall be submitted for the test of each particular slate under consideration.

Preparation of Specimens. 9. The slate for test shall have been split to a thickness of approximately $\frac{5}{8}$ in. and sawed into strips 12 in. in length by 4 in. in width. Half of these shall be cut with the length parallel to the grain and the other half with the length perpendicular to the grain. The 12 by 4-in. faces shall be planed to smooth surfaces and rubbed down to a thickness of approximately $\frac{3}{8}$ in., care being taken to have the finished surfaces as nearly parallel as practical.

IV. PROCEDURE

Drying, Measuring and Testing. 10. The specimens shall be dried, measured and placed in a testing machine of the same type as described for the modulus of rupture test. The specimens shall be supported flatwise on knife edges of the type shown in Fig. 1, spaced 10 in. apart and loaded at the center by means of a third knife edge at the center of the span. A deflectometer, capable of measuring accurately to 0.001 in., shall be used to indicate the deflections at the center of the span. The load shall then be applied in 50-lb. increments and the corresponding deflections recorded.

Computing Results. 11. The load deformation readings shall be plotted on cross-section paper to a convenient scale, and a straight line drawn to represent as nearly as possible the average of the points plotted. If this line does not pass through the origin of coordinates, a corrected line parallel to this line shall be drawn through the origin. The modulus of elasticity shall be computed from the coordinates of some convenient point on the corrected curve from the formula:

$$E=\frac{Wl^3}{4\Delta bd^3}$$

where w = the load ordinate of this point, Δ = deformation ordinate of same, l = the length of span in inches, b = the width of the specimen in inches, and d = the thickness of the specimen in inches.

Reporting Results. 12. (*a*) The average value of the three specimens cut with the length parallel to the grain shall be reported as the modulus of elasticity parallel to the grain, and the average of the other three, as the modulus of elasticity perpendicular to the grain.

(*b*) In case one specimen shows a value 25 per cent lower than the average of its group, it shall be examined for defects, and if the low result appears to be due to an imperfection, the average of the other two determinations shall be reported as the modulus of elasticity of the group. All determinations shall be reported as information.

TENTATIVE SPECIFICATIONS
FOR
ACID-RESISTING ASPHALT MASTIC[1]

Serial Designation: D 223 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope.

1. (*a*) These specifications cover the materials for asphalt mastic suitable for use in waterproofing, consisting of mastic cake, asphalt cement and mineral aggregate.

(*b*) The grade of asphalt mastic covered by these specifications is suitable for waterproofing, tank linings, floors, etc., where dilute mineral acids at temperatures below 100° F. (38° C.) are encountered.

Properties.

2. This asphalt is a mixture of mastic cake, asphalt cement and mineral aggregate, which at a temperature of approximately 205° C. (400° F.) is sufficiently plastic to be poured into place and compressed with a wooden trowel or equivalent tool into a compact mass.

As used for strictly waterproofing purposes the mastic cake, asphalt cement and mineral aggregate are combined under heat and manual stirring in substantially the following proportions by weight:

Mastic Cake	48 per cent
Asphalt Cement	5 "
Mineral Aggregate	47 "

Mastic Cake.

3. (*a*) The mastic cake shall conform to the following requirements:

Weight per cake	not more than 80 lb.
Soluble in pure benzol	14 to 18 per cent by weight
Insoluble in pure benzol	82 to 86 per cent by weight

(*b*) The matter soluble in pure benzol shall be asphalt cement, which, after recovery from the mastic cake, shall conform to the requirements of the Tentative Specifications for Asphalt Cement, 25

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

to 30 Penetration, for Use in Sheet Asphalt and Asphaltic-Concrete Pavements (Serial Designation: D 163 – 23 T) of the American Society for Testing Materials.[1]

(*c*) The matter insoluble in pure benzol shall be granular mineral matter which after recovery from the mastic cake shall conform to the following requirements as to grading.

Passing 200-mesh sieve	not less than 25 per cent
Passing 10-mesh sieve, retained on 50-mesh sieve	not more than 25 "
Retained on 10-mesh sieve	not more than 1 "

It shall contain not more than 5 per cent by weight of material soluble in hydrochloric acid.

Asphalt Cement. 4. The phalt cement shall conform to the requirements of the Tentative Specifications for Asphalt Cement, 25 to 30 Penetration, for Use in Sheet Asphalt and Asphaltic-Concrete Pavements (Serial Designation. D 163 – 23 T) of the American Society for Testing Materials.[1]

Mineral Aggregate. 5. The mineral aggregate shall be gravel or crushed stone and sand and free from carbonates. It shall conform to the following requirements as to grading:

Passing 200-mesh sieve	none
Passing 50-mesh sieve, retained on 200-mesh sieve	nor more than 25 per cent
Passing 10-mesh sieve, retained on 50-mesh sieve	not less than 25 "
Passing 4-mesh sieve, retained on 10-mesh sieve	not less than 50 "
Retained on 4-mesh sieve	not more than 10 "

It shall contain not more than 5 per cent by weight of material soluble in hydrochloric acid.

Method of Testing. 6. The properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147 – 25 T) of the American Society for Testing Materials.[2]

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[1] See p. 454.
[2] See p. 622.

TENTATIVE SPECIFICATIONS

FOR

SMOOTH-SURFACED ASPHALT ROLL-ROOFING[1]

Serial Designation: D 224 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. These specifications cover smooth-surfaced roll-roofing (medium and heavy weights), either 36 in. or 32 in. in width, composed of roofing felt saturated and coated on both sides with asphalt, and surfaced top and bottom with powdered talc or mica. The medium weight roofing is intended for use on semi-permanent structures and the heavy weight roofing for use on permanent structures.

I. MANUFACTURE

Manufacture. 2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant, then coated on both sides with an asphaltic coating, and finally surfaced on both sides with powdered talc or mica.

Character of Felt. 3. The felt shall be a roofing felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth, and upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Character of Saturant and Coatings. 4. The saturant and coatings shall be composed principally of asphaltic materials, and the presence of saponifiable constituents shall be permitted.

Character of Felt after Saturation. 5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Character of Mineral Surfacings. 6. The surfacing shall be non-combustible material such as powdered talc or mica.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

II. PHYSICAL PROPERTIES

7. The fabricated product shall conform to the following requirements:

(*a*) *Width of Roll.*—Either 36 or 32 in., as may be specified, $\pm\frac{1}{4}$ in.

(*b*) *Area of Roll.*—The average area of the rolls examined shall contain as specified not less than either 108 sq. ft. or 216 sq. ft., which shall be sufficient to cover one or two "squares," respectively.

(*c*) *Weight of Wrapping, Packing, Nails and Cement per "Square,"* maximum, 3 lb.

NOTE.—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and seller, in which event the above weight may be increased.

(*d*) *Weight of Material per 108 sq. ft. Exclusive of Wrapping, Packing, Loose Mineral Surfacing, Nails and Cement.*—

Medium weight, average, 42 lb., minimum, 40 lb.
Heavy weight, average, 52 lb., minimum, 49 lb.

(*e*) *Pliability at 77° F. (25° C.).*—Shall not crack on 10-mm. mandril.

(*f*) *Behavior on Heating to 176° F. (80° C.) for 2 Hours.*—Volatile matter, maximum, 1.5 per cent. There shall be no flowing, sagging, blistering or absorption of the surface coatings.

(*g*) *Saturant in Moisture-free Felt.*—Medium and heavy weights, minimum, 140 per cent by weight.

(*h*) *Weight of Moisture-free Desaturated Felt per 108 sq. ft.*—

Medium weight, minimum, 10.8 lb. ("Number" 48).
Heavy weight, minimum, 13.5 lb. ("Number" 60).

(*i*) *Ash in Moisture-free Desaturated Felt.*—Maximum, 10 per cent.

(*j*) *Weight of Mineral Surfacings and Mineral Matter in Coatings per 108 sq. ft.*—Medium and heavy weights, maximum, 2 lb.

NOTE.—Should the weight exceed 2 lb. the material shall nevertheless be acceptable, provided the finished weights per 108 sq. ft. are increased by a like amount over the figures specified in Paragraph (*d*).

III. WORKMANSHIP AND FINISH

Finished Appearance. 8. The surfaces shall be smooth or veined. Deeply ribbed surfaces shall not be acceptable.

Freedom from Defects. 9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

10. The coating and mineral surfacing shall be applied uniformly up to the edges of the sheet. **Distribution of Coatings and Surfacing.**

11. The mineral surfacing shall uniformly cover both sides of the sheet. **Adhesion of Surfacing.**

12. The coating and surfacing shall be of such a nature that the finished product will not crack, or be sticky enough to cause tearing or material damage, upon being unrolled at atmospheric temperatures above 50° F. (10° C.). **Stickiness.**

IV. PACKING

13. (*a*) The roofing shall be put up in rolls of 108 sq. ft. or 216 sq. ft. as specified in the order. No roll shall contain more than two pieces of roofing, nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the rolls shall be protected with metal caps, cloth or other suitable means. The nails and lap-cement shall be furnished either in each roll, or in bulk as specified in the order. When furnished in bulk, the cloth or metal ends may be omitted. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed upon by the purchaser and seller and shall contain printed directions for laying. Rolls for export shipment shall be packed as agreed upon by the purchaser and seller.

(*b*) *Nails.*—The gage of the wire shall not be greater than "A. S. & W." No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall not be less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. thick; the shank shall be between $\frac{3}{4}$ and 1 in. long, with its lower end pointed, and shall be either smooth or barbed, and if the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted. There shall be furnished per "square" not less than an average of 252 nails for 36-in., and not less than an average of 275 nails for 32-in. roofing.

(*c*) *Lap-cement.*—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of 71° F. (21.5° C.) when tested by the Tagliabue Closed-cup Tester. There shall be furnished for each square of 36-in. roofing not less than $\frac{3}{4}$ pint and for 32-in.

roofing not less than 1 pint. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased by $\frac{1}{4}$ pint per "square."

V. SAMPLING AND TESTING

14. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles (Serial Designation: D 228 – 25 T) of the American Society for Testing Materials.[1]

VI. INSPECTION

15. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 628.

TENTATIVE SPECIFICATIONS
FOR
SLATE-SURFACED ASPHALT ROLL-ROOFING AND SLATE-SURFACED ASPHALT SHINGLES[1]

Serial Designation: D 225 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

Scope. 1. These specifications cover slate-surfaced roll roofing, either 36 or 32 in. in width, and slate-surfaced composition shingles, composed of roofing-felt saturated and coated on both sides with asphalt and surfaced on the weather side with granulated slate (green, red, black, or such color or combinations of color as may be agreed upon between the purchaser and seller), or similar mineral material to be specified by the purchaser, and on the reverse side with powdered talc or mica to prevent the roofing or shingles adhering together in the package. Such roofing and shingles are intended for use on semi-permanent and permanent buildings.

I. MANUFACTURE

Manufacture. 2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant, then coated on both sides with an asphaltic coating, and finally surfaced on the weather side with granulated slate or similar mineral material to be specified by the purchaser, and on the reverse side with powdered talc or mica.

Character of Felt. 3. The felt shall be a roofing felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth, and upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

Character of Saturant. and Coatings. 4. The saturant and coatings shall be composed principally of asphaltic materials, and the presence of saponifiable constituents shall be permitted.

Character of Felt after Saturation. 5. The felt shall be thoroughly and uniformily saturated, and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Character of Mineral Surfacing. 6. The surfacing on the weather side shall be granular crushed slate or similar mineral material, as may be specified by the purchaser (in either green, red, blue-black or such other color or combinations of color as may be agreed upon between the purchaser and seller), shall be sufficiently free from fines or dust to permit the firm adhesion of the larger particles of the surfacing and provide a uniform distribution. The under side of the sheet shall be surfaced with a non-combustible material such as powered mica or talc.

II. PHYSICAL PROPERTIES

7. The fabricated product shall conform to the following requirements:

(a) *Width of Roll; Size and Form of Shingles:* Slate roll-roofing shall be either 36 or 32 in. in width, as may be specified, $\pm \frac{1}{4}$ in.

Shingles shall be of a size and form as may be agreed upon between the purchaser and seller.

(b) *Area per Package: Slate-Roll.*—The average area of the rolls examined shall contain not less than 108 sq. ft., which shall be sufficient to cover one "square".

Shingles.—Sufficient material shall be furnished per "square" as may be agreed upon between the purchaser and seller to adequately waterproof, when laid, the surface to which the shingles are applied.

(c) *Weight of Wrapping and Packing Material per "Square":* Slate Roll including nails and lap-cement, maximum, 3 lb.

NOTE.—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and seller, in which event the above weights may be increased.

Shingles (packing only), maximum 12 lb.

(d) *Weight of Material per 108 sq. ft. Exclusive of Wrapping, Packing, Loose Mineral Surfacing, Nails and Cement.*—Slate Roll and Shingles, average, 82 lb., minimum, 80 lb.

(*e*) *Pliability at 77° F. (25° C.): Slate Roll.*—Shall not crack on 25-mm. mandril. *Shingles.*—No requirements.

(*f*) *Behavior on Heating to 176° F. (80° C.) for 2 hours.*—Volatile matter, maximum 1.5 per cent. There shall be no flowing, sagging, blistering or absorption of the surface coatings. Granular surfacings shall not slide more than $\frac{1}{16}$ in. when suspended vertically.

(*g*) *Saturant in Moisture-Free Felt:*
Slate Roll, minimum, 140 per cent by weight.
Shingles, minimum, 150 per cent by weight.

(*h*) *Weight of Moisture-Free Desaturated Felt per 108 sq. ft.*—Slate roll and shingles, minimum 10.8 lb. ("Number" 48).

(*i*) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

(*j*) *Weight of Mineral Surfacings and Mineral Matter in Coating per 108 sq. ft.*—Slate roll and shingles, maximum 35 lb.

III. WORKMANSHIP AND FINISH

Finished Appearance. 8. The weather surface shall be uniform in finish and texture.

Freedom from Defects. 9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Distribution of Coatings and Surfacings. 10. The coating and granules shall be applied in a uniformly smooth layer over the entire weather surface, except that slate-roll roofing may be provided with a bare lapping edge, 2 in. wide, along one edge of the sheet, where either the slate alone, or both the slate and coating may be omitted. The under surface of the sheet shall have the asphalt coating and mineral surfacing applied uniformily to the edges.

Adhesion of Surfacing. 11. The granular surfacing shall be embedded in the coating in such a manner that when rubbed vigorously with the hand, sufficient surfacing will remain embedded to completely cover the coating.

Stickiness. 12. For slate-roll-roofing, the coating and surfacings shall be of such a nature that the finished product will not crack or stick together so as to cause tearing or damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.). For shingles, the coating and surfacings shall be of such a nature that the finished products will not adhere together in the bundles so as to tear off or loosen the coating when unpacked.

IV. PACKING

13. (*a*) Slate roll-roofing shall be put up in rolls of 108 sq. ft. No rolls shall contain more than two pieces nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the roll shall be protected with metal caps, cloth or other suitable means. The nails and lap-cement shall be furnished either in each roll or in bulk, as specified in the order. When furnished in bulk, the cloth or metal ends may be omitted. Rolls for export shipment shall be packed as agreed upon by the purchaser and seller.

Shingles shall be packed in bundles weighing not more than 135 lb. gross. The bundles shall either be packed in cardboard cartons or between wooden or cardboard ends and tied with rope, wire or steel bands so as to make a substantial package, as may be agreed upon by the purchaser and seller. Each roll or package shall be plainly marked with the manufacturer's name and brand or as agreed upon by the purchaser and seller, and shall contain printed directions for laying.

(*b*) *Nails.*—The gage of the wire shall not be greater than "A. S. & W." No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall be not less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. thick; the shank shall be between $\frac{3}{4}$ and 1 in. long for slate-roll roofing, and for shingles 1 in. long when laid on solid sheathing, or $1\frac{1}{2}$ to $1\frac{3}{4}$ in. when laid over old shingles. The lower end of the shank shall be pointed, and either smooth or barbed, and if the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted. There shall be furnished per "square" of slate-roll roofing not less than an average of 252 nails for 36 in., and not less than an average of 275 nails for 32-in. roofing. For shingles, the nails shall be purchased separately, and the number required per "square" will depend upon the size and form of the shingles and shall be agreed upon by the purchaser and seller.

(*c*) *Lap-cement* (*for Slate Roll.*)—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of

71° F. (21.5° C.) when tested by the Tagliabue Closed-cup Tester. There shall be furnished for each square of 36-in. roofing not less $\frac{3}{4}$ pint and for 32-in. roofing not less than 1 pint. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased $\frac{1}{4}$ pint per "square".

V. SAMPLING AND TESTING

14. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Smooth-Surfaced Asphalt Roll-Roofing, Slate-Surfaced Asphalt Roll-Roofing and Slate-Surfaced Asphalt Shingles (Serial Designation: D 228 – 25 T) of the American Society for Testing Materials.[1]

VI. INSPECTION

15. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 628.

TENTATIVE SPECIFICATIONS

FOR

ASPHALT-SATURATED ROOFING FELT FOR USE IN WATERPROOFING AND IN CONSTRUCTING BUILT-UP ROOFS[1]

Serial Designation: D 226 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope.

1. These specifications cover bituminized felted fabrics, either 36 or 32 in. in width, composed of roofing felt saturated, but not coated, with asphalt for use in the membrane system of waterproofing and in the construction of built-up roofs.

I. MANUFACTURE

Manufacture.

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with an asphaltic saturant.

Character of Felt.

3. The felt shall be a roofing-felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth, and upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Character of Saturant.

4. The saturant shall be composed of asphaltic materials.

Character of Felt after Saturation.

5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2 in. strips at random across the entire sheet and splitting them open for their full length.

Surfacing.

6. The surface of the fabric shall not be coated or covered with talc or other substance which would tend to interfere with the adhesion between the fabric and the plying cement.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

II. PHYSICAL PROPERTIES

7. The fabricated bituminized felt shall conform to the following requirements: **Fabricated Bituminized Felt.**

(a) *Width of Roll.*—Either 36 or 32 in., as may be specified, $\pm\frac{1}{4}$ in.

(b) *Gross Weight per Roll.*—40 to 80 lb.

(c) *Weight of Wrapping and Packing Material per Roll.*—Maximum $\frac{1}{2}$ lb.

(d) *Weight of Material per 100 sq. ft. exclusive of Packing and Wrapping.*—14 lb. ± 1 lb.

(e) *Moisture.*—Maximum, 2.5 per cent of the net weight.

(f) *Loss on Heating (Exclusive of Moisture) at 105° C. (221° F.) for 5 hours.*—Maximum, 2 per cent.

(g) *Pliability at 77° F. (25° C.).*—Shall not crack when bent flat on itself.

(h) *Saturant in Moisture-Free Felt.*—Minimum 140 per cent by weight.

8. The desaturated felt shall conform to the following requirements: **Desaturated Felt.**

(a) *Weight of Moisture-Free Desaturated Felt.*—The average "Number" (expressed on the basis of pounds per 480 sq. ft.) shall be 27, equivalent to 5.625 lb. per 100 sq. ft., with a permissible variation of 10 per cent.

(b) *Average Thickness of Desaturated Felt.*—Minimum, 0.025 in.

(c) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

III. WORKMANSHIP AND FINISH

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations. **Freedom from Defects.**

10. The rolls shall not crack, or be sticky enough to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.). **Stickiness.**

IV. PACKING

11. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent

it shifting from position. The ends of the roll need not be covered. As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the purchaser and seller. No roll shall contain more than two pieces, and there shall not be more than 3 per cent of rolls containing two pieces in any shipment. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed by the purchaser and seller, and shall bear a notation showing the type of saturant.

V. SAMPLING AND TESTING

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing (Serial Designation: D 146 – 25 T) of the American Society for Testing Materials.[1]

VI. INSPECTION

13. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

[1] See p. 612.

TENTATIVE SPECIFICATIONS
FOR
COAL-TAR SATURATED ROOFING FELT FOR USE IN WATERPROOFING AND IN CONSTRUCTING BUILT-UP ROOFS[1]

Serial Designation: D 227 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. These specifications cover bituminized felted fabrics, either 36 or 32 in. in width, composed of roofing felt saturated, but not coated, with refined coal tar, for use in the membrane system of waterproofing and in the construction of built-up roofs.

I. MANUFACTURE

Manufacture. 2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a coal-tar saturant.

Character of Felt. 3. The felt shall be a roofing-felt produced by "felting" vegetable and animal fibers. The surface of the felt shall be uniformly smooth, and upon splitting or tearing on the bias, shall appear reasonably free from lumps of underbeaten stock (*i.e.*, stock which has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (*i.e.*, fragments of stone, metal, leather, rubber, straw, wood, etc.).

Character of Saturant. 4. The saturant shall be composed of coal tar from which the highly volatile constituents have been removed.

Character of Felt after Saturation. 5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surfacing. 6. The surface of the fabric shall not be coated or covered with talc or other substance which would tend to interfere with the adhesion between the fabric and the plying cement.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

II. PHYSICAL PROPERTIES

Fabricated Bituminized Felt.

7. The fabricated bituminized felt shall conform to the following requirements:

(a) *Width of Roll.*—Either 36 or 32-in., as may be specified, $\pm\frac{1}{4}$ in.

(b) *Gross Weight per Roll.*—40 to 80 lb.

(c) *Weight of Wrapping and Packing Material per Roll.*—Maximum, $\frac{1}{2}$ lb.

(d) *Weight of Material per 100 sq. ft. exclusive of Packing and Wrapping.*—14 lb. ± 1 lb.

(e) *Moisture.*—Maximum, 2.5 per cent of the net weight.

(f) *Distillate*, per cent by weight to 210° C., calculated on extracted saturant, not more than 2.

Distillate, per cent by weight to 235° C., calculated on extracted saturant, not more than 5.

(g) *Pliability at 77° F. (25° C.).*—Shall not crack when bent flat on itself.

(h) *Saturant in Moisture-Free Felt.*—Minimum, 140 per cent by weight.

Desaturated Felt.

8. The desaturated felt shall conform to the following requirements:

(a) *Weight of Moisture-Free Desaturated Felt.*—The average "Number" (expressed on the basis of pounds per 480 sq. ft.) shall be 27, equivalent to 5.625 lb. per 100 sq. ft., with a permissible variation of 10 per cent.

(b) *Average Thickness of Desaturated Felt.*—Minimum, 0.025 in.

(c) *Ash in Moisture-Free Desaturated Felt.*—Maximum, 10 per cent.

III. WORKMANSHIP AND FINISH

Freedom from Defects.

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Stickiness.

10. The rolls shall not crack, or be sticky enough to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50° F. (10° C.).

IV. PACKING

11. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner which will prevent it shifting from position. The ends of the roll need not be covered.

As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the purchaser and seller. No roll shall contain more than two pieces, and there shall not be more than 3 per cent of rolls containing two pieces in any shipment. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed by the purchaser and seller, and shall bear a notation showing the type of saturant.

V. SAMPLING AND TESTING

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing (Serial Designation: D 146 – 25 T) of the American Society for Testing Materials.[1]

VI. INSPECTION

13. Inspection of material shall be made as agreed upon by the purchaser and seller as part of the purchase contract.

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[1] See p. 612.

TENTATIVE METHODS OF TESTING FELTED AND WOVEN FABRICS SATURATED WITH BITUMINOUS SUBSTANCES FOR USE IN WATERPROOFING AND ROOFING[1]

Serial Designation: D 146 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1922; Revised, 1923, 1925.

Scope. 1. These methods cover the examination of felted or woven fabrics saturated (but not coated) with asphalt or coal-tar products, for use in the "membrane" system of waterproofing.

I. SAMPLING.

Sampling. 2. From each shipment or portion thereof representing a product of the same kind, class and weight, a number of rolls shall be selected at random, equivalent to the cube root of the total number of rolls in the lot. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes:

Packages in Shipment.	Number of Packages Selected.	Packages in Shipment.	Number of Packages Selected.
2 to 8	2	1332 to 1728	12
9 to 27	3	1729 to 2197	13
28 to 64	4	2198 to 2744	14
65 to 125	5	2745 to 3375	15
126 to 216	6	3376 to 4096	16
217 to 343	7	4097 to 4913	17
344 to 512	8	4914 to 5832	18
513 to 729	9	5833 to 6859	19
730 to 1000	10	6860 to 8000	20
1001 to 1331	11	8001 to 9261	21

II. EXAMINATION OF FINISHED FABRIC.

Width. 3. The width of each roll taken shall be measured to the nearest $\frac{1}{32}$ in. The minimum, maximum and average width so obtained shall be reported.

Gross Weight per Roll. 4. Each roll taken shall be weighed intact to the nearest $\frac{1}{4}$ lb. and the minimum, maximum and average gross weight shall be reported.

[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

5. Each roll sampled shall be stripped. All the wrappers and packing material shall be weighed together to the nearest $\frac{1}{4}$ lb. and the average weight shall be reported. Each roll should be temporarily fastened with a piece of light string and handled carefully, so as not to disturb any detached surfacing material. **Wrapping and Packing Materials.**

6. The minimum, maximum and average net weight per roll shall be calculated by subtracting the average weight of the wrapping and packing material as determined in Section 5 from the average gross weight as determined in Section 4. **Net Weight per Roll.**

7. Each roll sampled shall be laid flat, the first convolution or two carefully unwound, and with a knife and straight edge the sheet shall be cleanly cut across at right angles to the edges. A section measuring 30 in. in the direction of the roll's length shall be removed. If the material is surfaced with sand or other fine material any detached particles belonging to each 30-in. section should be retained. The width of each section shall be measured to the nearest $\frac{1}{32}$ in. Each section, together with any detached surfacing, shall be weighed to the nearest gram. The weight in pounds per 100 sq. ft. or the weight in ounces per sq. yd. shall be calculated from the formulas: **Weight per Unit Area.**

$$\text{Pounds per 100 sq. ft.} = 1.0582 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

$$\text{Ounces per sq. yd.} = 1.5238 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

From the results obtained for each roll sampled, the minimum, maximum, and average weight shall be calculated.

NOTE.—As a referee method or in case any dispute arises regarding the weight "per unit area," the method described in Section 8 shall be followed. From the net weight of each roll determined as in Section 6, the weight per unit area may be calculated as a check.

8. The average area per roll in square feet shall be calculated by dividing the average net weight per roll, as determined in Section 6, by the average weight in pounds per 100 sq. ft. as determined in Section 7, and multiplying by 100. **Area per Roll.**

NOTE.—As a referee method or in case any dispute arises regarding the "area per roll" as calculated, each roll sampled shall be unwound and the total length measured within $\frac{1}{4}$ in. with a steel tape. From the width of each roll, as determined in Section 3, the "area per roll" may be accurately calculated as a check and the minimum, maximum and average for the shipment calculated.

9. If the material is surfaced with sand or other finely comminuted material, the surfacing shall be swept with a moderately stiff brush from all the 30-in. sections taken. All the material thus removed shall be caught and weighed to the nearest ounce. From the aggregate areas of all the sections taken, the average weight of detached comminuted surfacing shall be calculated in pounds per 100 sq. ft. **Detached Comminuted Surfacing.**

Thickness. 10. The thickness of each specimen shall be measured at 10 equally spaced points, 1 in. from a cut edge, with a micrometer gage having flat bearing surfaces at both contact points of not less than ½ in. in diameter and preferably of the spring-and-dial type. The readings for each roll sampled shall be averaged and the minimum, maximum and average thickness calculated to the nearest 0.001 in.

Moisture. 11. From each specimen, 2-in. strips shall be cut as shown at *A-1* and *A-2* in Fig. 1. The 2-in. strips shall be cut into 1-in. squares. About 50 g., selected at random, shall be accurately weighed and

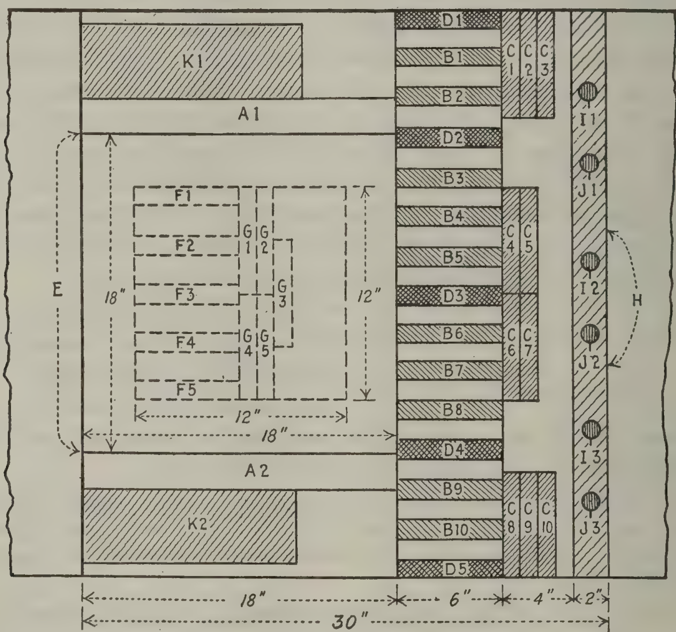

FIG. 1

distilled with 100 cc. of a coal-tar distillate of which 5 to 10 per cent boils below 100° C. (212° F.) and of which at least 90 per cent distills up to 180° C. (356° F.) in an apparatus of the form and under the conditions prescribed in the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (Serial Designation: D 95) of the American Society for Testing Materials.[1] The flask containing the solvent and sample shall be heated in a paraffin bath at a temperature of 302 to 338° F. (150 to 170° C.) until no further water passes over. The distillate shall be allowed to stand in the graduated receiver until the water separates, whereupon the volume of water shall be measured and its weight calculated. From this, the

[1] 1924 Book of A.S.T.M. Standards.

average percentage of moisture in the fabric, as received, shall be calculated.

12. (*a*) *Felted Fabrics.*—With a photographic trimmer having a stop attached exactly 1 in. behind the blade, ten strips shall be cut with the fiber grain, as shown at *B-1* to *B-10* (Fig. 1) and ten strips across the fiber grain as shown at *C-1* to *C-10*. Each strip should measure 6 in. and have parallel sides 1 in. apart (within $\frac{1}{32}$ in.) with the edges cut straight and clean. Both sets of strips shall be tested at 70° F. (21.1° C.), using a tension testing machine such as the Scott Strength Tester (Type F, 100 lb. capacity), the Perkins Strip Tester, or equal, in which the clamps are attached to swivels free to move in any direction. The test strips shall be gripped $1\frac{1}{2}$ in. on each end, leaving approximately 3 in. between the clamps. The tension shall be increased at a uniform speed of 3 lb. per second, using a metronome. If any strip breaks nearer than $\frac{1}{2}$ in. to either clamp, the reading shall be disregarded, and an additional strip shall be tested in its place. The ten readings, with and across the grain, respectively, shall be averaged for each roll sampled. From these results the average strength with and across the fiber grain, respectively, of the fabric as supplied, shall be calculated. **Strength.**

(*b*) *Woven Fabrics*—Woven fabrics shall be tested at 70° F. (21.1° C.) in accordance with the "grab test No. 2" as described in Section 12 (*b*) of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1]

NOTE.—As a referee method, or in case any dispute arises regarding the strength, the test shall be repeated, with the exception that the fabric before testing shall be exposed at least two hours in an atmosphere of 65 per cent relative humidity at 70° F. (21.1° C.).

13. With the trimmer described in Section 12 (*a*), five 6-in. strips shall be cut with the fiber grain, as shown at *D-1* to *D-5* (Fig. 1) and immersed in water at 77° F. (25° C.) for from 10 to 15 minutes. These strips shall be bent through 180 deg. at a uniform speed, in exactly 2 seconds, around a mandril, the diameter of which shall be as follows: The first, around a mandril 25 mm. in diameter; the second, around a mandril 20 mm. in diameter; the third, around a mandril 15 mm. in diameter; the fourth, around a mandril 10 mm. in diameter, and the fifth, around a mandril 5 mm. in diameter. The test may also be made at 32° F. (0° C.). The pliability shall be expressed numerically from 1 to 10 as follows: **Pliability.**

[1] 1924 Book of A.S.T.M. Standards.

(1) Cracks entirely through the sheet on the 25-mm. mandril.
(2) Cracks part way through the sheet on the 25-mm. mandril.
(3) Cracks on the 25-mm. mandril.
(4) Cracks on the 20-mm. mandril.
(5) Cracks on the 15-mm. mandril.
(6) Cracks on the 10-mm. mandril.
(7) Cracks on the 5-mm. mandril.
(8) Cracks when bent through 180 deg. flat on itself.
(9) May be bent through 180 deg. flat on itself in one direction without cracking, but will crack when bent through 360 deg. in the opposite direction.
(10) May be bent through 180 deg. flat on itself in one direction and then through 360 deg. flat on itself in the opposite direction without cracking.

The readings for each roll shall be averaged separately, and from these results the minimum, maximum and average pliability of the felt shall be calculated.

Water Absorbed. 14. (*a*) A section 18 in. square (as shown at *E*, Fig. 1) shall be cut from each specimen, weighed, and completely immersed in distilled water at 77° F. (25° C.) for 24 hours. The specimen shall then be removed and dried superficially by pressing lightly between two towels. As the moisture enters through the edges of the sheet more rapidly than through the surfaces, each specimen shall be trimmed to exactly 12 in. square, representing four-ninths of the original area, and reweighed rapidly. The increase in weight shall be calculated on the basis of the original test specimen by multiplying by $2\frac{1}{4}$. The percentage increase in weight represents the water absorption. The minimum, maximum and average for the shipment shall be calculated.

(*b*) Five strips shall be cut with the fiber grain as shown at *F 1* to *F 5* (Fig. 1); five other strips shall be cut across the fiber grain as shown at *G 1* to *G 5* (Fig. 1) and the strength shall be redetermined as in Section 12. The decrease in strength in percentage shall be calculated after the specimen has been subjected to water.

Loss on Heating. 15. (*a*) From each specimen, 12 by 6-in. strips shall be cut at *K-1* and *K-2* in Fig. 1, care bring taken not to disturb any of the detached surfacing. Each strip shall be weighed and suspended in the center of an air oven maintained at 221° F. (105° C.) ± 5° F. (3° C.) with a thin wire fastened through holes punctured near one edge. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The strips shall be kept in the oven for exactly 5 hours, then cooled and removed carefully, and each specimen weighed. The average loss shall be

calculated as a percentage and the average percentage of moisture (ascertained in Section 11) deducted. The final figure shall represent the average loss on heating, exclusive of moisture.

(*b*) *Coal-Tar Saturated Felt Only.*—From the rolls making up the sample, strips 6 in. wide shall be cut across the sheet. Sufficient of these shall be taken to make up a sample of 250 g. ±5.0 g. These strips then shall be rolled up and placed in the wire basket of the extraction apparatus described in the Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147 – 25 T) of the American Society for Testing Materials[1] or other extraction apparatus of suitable construction. The sample shall then be covered with a disk of soft filter paper to insure an even distribution of the solvent. The extraction shall be carried out as described in Methods D 147 – 25 T. **Extraction of Saturant.**

(*c*) The CS_2 extract containing the bitumen shall be transferred to a 500-cc., short-neck, round-bottom flask. The flask shall be equipped with a Hempel column, 16 cm. in length, 15 mm. in inside diameter and filled to a depth of 5 cm. with glass beads. The Hempel column shall be connected with a water cooled condenser. A 0 – 80° C. thermometer as described in the Tentative Method of Float Test for Bituminous Materials (Serial Designation: D 139 – 25 T) of the American Society for Testing Materials[2] shall be placed in the Hempel column so that the top of the bulb shall be opposite the bottom of the tubulature. Distillation for the removal of CS_2 shall be continued by means of a steam bath until drops cease to fall from the condenser. The distillation then shall be carried out over a small flame, heating carefully and observing the thermometer until the distillation ceases and the temperature of the vapor as indicated by the thermometer shows no further rise on slightly increasing the flame. **Recovery of Saturant from CS_2 Extract.**

(*d*) When the contents of the flask is sufficiently cool to be poured without appreciable vaporizing, 100 g. ±0.1 g. shall be distilled in accordance with the method described in Sections 10, 11 and 12 of the Standard Methods of Sampling and Analysis of Creosote Oil (Serial Designation: D 38) of the American Society for Testing Materials.[3] **Distillation of Saturant.**

Fractions shall be taken at 210° C. and 235° C. and the distillation stopped at that point. The per cent of distillate by weight at each temperature shall be calculated on the basis of the weight of sample taken for distillation.

III. DESATURATING THE FABRIC.

16. From each roll sampled, a 2-in. strip (within $\frac{1}{32}$ in.) shall be cut across the specimen as shown at *H* (Fig. 1). Each strip shall be extracted with c. p. benzol in an extractor described in Section 4 of **Desaturated Fabric.**

[1] See p. 622. [2] See p. 503. [3] 1924 Book of A.S.T.M. Standards.

the Tentative Methods of Testing Bituminous Mastics, Grouts and Like Mixtures (Serial Designation: D 147 – 24 T) of the American Society for Testing Materials,[1] the extraction being continued for several hours after the drippings have become colorless. The desaturated fabric shall be removed and heated in a ventilated oven at 225° F. (107.1° C.), cooled in a desiccator, any adhering comminuted surfacing being brushed off and retained, and weighed as rapidly as possible. The heating shall be repeated until the weight of the fabric remains constant as determined by two consecutive weighings taken not less than ten minutes apart which shall show a further loss of not more than 0.1 per cent. This will give the weight of the fabric in the moisture-free state. Where a coal-tar pitch saturant has been used, the moisture-free weight of the desaturated fabric shall be corrected for the carbonaceous matter retained mechanically in its interstices, by means of the method described in Section 17.

Retained Carbonaceous Matter.

17. The following colorimetric method shall be used to ascertain the carbonaceous matter derived from a coal-tar pitch saturant and retained by the desaturated fabric:

(*a*) About 15 g. of an unsaturated fabric of the same general character as the one under examination, shall be macerated by boiling in water, disintegrating with a rotary egg-beater and picking the fibers apart with needles. The fibers shall be filtered through fine cloth and dried at a temperature of 225° F. (107.1° C.) to constant weight. One gram of the fibers shall be accurately weighed into a flask and diluted to exactly 100 cc. with distilled water at room temperature. About 50 g. of glass beads shall be added and the contents of the flask shaken vigorously until the fibers are reduced to a homogeneous pulp in uniform suspension.

(*b*) A distilled coal-tar, having approximately 10 to 25 per cent of insoluble carbonaceous matter, shall be procured and the carbonaceous matter extracted with benzol until it is free from soluble matter; then dried at a temperature of 225° F. (107.1° C.) to constant weight. One gram of the purified carbonaceous matter shall be accurately weighed and diluted to exactly 100 cc. at room temperature with a starch solution of a consistency sufficient to carry the carbonaceous matter into temporary suspension. (A 12.5-per-cent solution is recommended.)

(*c*) The liquid carrying the fibers, obtained as described in Paragraph (*a*), shall be titrated with the suspension of carbonaceous matter, obtained as described in Paragraph (*b*), and from time to time a field prepared from a drop of the well agitated mixture shall be examined

[1] See p. 622.

under a microscope at 100 diameters magnification until the color exactly matches a field prepared from the desaturated fabric under examination (obtained as described in Section 16), when both are viewed side by side under parallel conditions. The end-point is fairly sharply defined. The burette reading gives directly the percentage of carbonaceous matter adhering to the moisture-free fabric under examination.

(*d*) The weight of moisture-free fabric ascertained in Section 16 shall be corrected by deducting the weight of adhering carbonaceous matter.

Total Comminuted Surfacing.

18. The total comminuted surfacing plus any filler present in the bituminous saturant represents the sum of the detached comminuted surfacing (Section 9), the amount brushed off from the surface of the desaturated fabric (Section 16), plus the amount recovered upon evaporating and igniting the bituminous extract obtained in accordance with Section 16.

Bituminous Saturant.

19. The weight of bituminous saturant represents the difference between the weight of the original fabric (Section 7), and the sum of the weights of the moisture-free desaturated fabric (Sections 16 and 17), plus moisture (Section 11), plus the total comminuted surfacing and filler present (Section 18).

IV. EXAMINATION OF THE EXTRACTED FABRIC.

(*A*) *Woven Fabrics.*

Weight.

20. Woven fabrics shall be tested for weight and number of threads per in. in accordance with Sections 6 to 11, inclusive, of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1]

(*B*) *Felted Fabrics.*

Weight.

21. The weight in pounds per 480 sq. ft. of the desaturated felt (corrected for any carbonaceous matter present) for each specimen examined, shall be calculated separately from the formulas:

$$\text{“Number”} = \frac{\text{Weight of specimen in grams}}{\text{Area of specimen in square centimeters}} \times 983$$

or

$$\text{“Number”} = \frac{\text{Weight of specimen in grams}}{\text{Area of specimen in square inches}} \times 152$$

This represents the "number" of the moisture-free felt on the felt makers' scale. The minimum, maximum and average "number" of the felt in its moisture-free state shall be calculated. This may be corrected to the condition in which it actually existed in the material

[1] 1924 Book of A.S.T.M. Standards.

under examination, by adding thereto the percentage of moisture as determined in Section 11.

Ash. 22. A representative sample shall be secured by cutting from each strip of desaturated felt a piece about $\frac{1}{2}$ in. in diameter as shown at *I-1*, *I-2*, and *I-3* (Fig. 1). About 25 g. selected at random from all the specimens sampled in this manner shall be accurately weighed, and incinerated in a weighed porcelain or quartz crucible either over an open flame or in a muffle, until all the carbon is consumed. A few drops of ammonium carbonate solution shall be added, the sample ignited gently, and weighed. The percentage of ash shall be calculated on the basis of the moisture-free felt.

Note.—As a referee method, or in case greater accuracy is desired, the three portions taken from each specimen roll shall be weighed and ignited separately. The minimum, maximum and average of ash present shall be calculated on the basis of the moisture-free felt.

Fibers Present. 23. (*a*) A representative sample shall be secured by cutting from each strip of desaturated felt a piece about $\frac{1}{2}$ in. in diameter as shown at *J-1*, *J-2*, and *J-3* (Fig. 1). The pieces shall be placed in a small beaker, boiled with water and macerated until the fibers are well separated and reduced to a pulp. They shall then be transferred to a stout flask. About 50 g. of glass beads shall be added and the contents of the flask shaken vigorously until the fibers are reduced to a homogeneous mass in a uniform suspension. A small portion of the well-mixed pulp shall be removed with a dropper or pipette and three or four drops shall be rapidly transferred to a microscope slide. Ten such slides shall be prepared, refilling the dropper each time.

(*b*) The drops shall be spread thinly over the microscope slides with a needle so that the fibers will be uniformly distributed and not bunched together. The slides shall then be placed in an air-bath to expel the water, cooled and stained with a drop or two of zinc-chlor-iodide solution.

Note—The zinc-chlor-iodide solution may be prepared by dissolving 4 g. of potassium iodide and 0.1 g. of iodine in 12 cc. of water, and then adding 20 g. of zinc chloride and allowing the liquid to stand until all undissolved matter has settled out. This solution must be kept in a dark place and prepared freshly from time to time, as it loses its strength. To test out the stain, a mixture of equal parts of rag filter paper, bleached soda pulp and bleacher sulfite pulp, shall be prepared, and tested as directed. If the stain is of the correct strength, the rag fibers will show a wine-red color, the soda pulp a dark purple color, due to the thicker and more opaque fiber walls, and the sulfite pulp a light purple color due to the thinner fiber walls. If the purple color is more of a violet, then too much iodine is present and more water or zinc chloride should be added. Zinc chloride intensifies the purple color, iodine the wine-red and yellow colors, and water serves to weaken the color that predominates.

(*c*) The stained pulp shall be covered with round or square pieces of thin glass approximately $\frac{1}{2}$ in. in diameter, and the excess of staining solution shall be removed from the edges with a piece of filter paper.

(*d*) The slides shall be examined under a microscope having a magnification of about 100 diameters.

(*e*) The various fibers are colored as follows:

Rag Fibers.—

Cotton and linen fibers—stained wine-red.

Wool fibers—unstained by the solution.

Jute and manila fibers—stained yellowish-brown.

Paper, Wood, Straw, etc., Fibers.—

Mechanical wood pulp, sawdust, straw, bark, etc.—stained lemon yellow.

Chemical wood pulp, (sulfite or soda), stained grayish purple to purple.

(*f*) The eye may be trained to the colors and shapes of the various fibers by examining specimens of known composition under similar conditions. In this way the fibers may readily be identified in unknown mixtures by:

(1) The colors to which they are stained.

(2) Their characteristic sizes and shapes.

(*g*) The fibers shall be counted in each field, disregarding any short ends projecting into the field or small fragments interspersed throughout it. Small particles of sizing, clay, or mineral matter should likewise be disregarded.

(*h*) Five fields shall be selected at random on each slide and the fibers counted in each. A total of 50 fields will be thus examined. The aggregate number of each variety of fiber counted in all the fields shall be calculated in percentage.

(*i*) The fibers present shall be reported in percentage *based on their count*, and should be generally accurate to within 5 per cent, depending upon the skill of the operator.

NOTE 1.—As the dimensions and specific gravity of the fibers have not been taken into consideration, the method is obviously inaccurate to this extent. Spence and Krauss[1] have worked out a set of factors for expressing the results into terms of weight, taking into consideration the dimensions and specific gravity of the fibers. This method is recommended, although it is somewhat slow and laborious.

NOTE 2.—As a referee method, or in case greater accuracy is desired, a separate microscope test shall be run on the three portions taken from each specimen roll. The minimum, maximum and average percentage of the various fibers present shall then be calculated.

[1] *Paper*, Vol. XX, No. 11, p. 11, May 23, 1917.

TENTATIVE METHODS OF TESTING BITUMINOUS MASTICS, GROUTS AND LIKE MIXTURES[1]

Serial Designation: D 147 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923, 1924, 1925.

I. DEFINITIONS.

Scope.

1. These methods apply to bituminous mastics, grouts and like mixtures, which may be classified and defined as follows:

Bituminous Grout.—A mixture of bituminous material as a binder and sandy mineral matter as an aggregate, which when heated to a suitable temperature becomes sufficiently fluid to flow into place without mechanical manipulation, and which on cooling congeals to a compact mass.

Asphalt Mastic.—(1) A mixture containing asphaltic material as a binder and graded mineral matter as an aggregate, or (2) pulverized native rock asphalt (to which asphaltic material may have been added); either of which when heated to a suitable temperature may be poured into place but which requires a trowel to form it into a compact mass.

Asphalt Mastic Cake.—A mixture containing asphaltic material as a binder and an aggregate consisting chiefly of calcareous or siliceous dust cast into the form of blocks or "cakes" and adapted for use in preparing asphalt mastic.

II. PREPARATION OF SAMPLES.

Preparation of Samples.

2. (*a*) Bituminous grouts shall be heated in an oven or on a hot plate in a pan or other suitable container at the lowest possible temperature to prevent overheating and volatilization, and when sufficiently fluid, shall be thoroughly stirred to insure a uniform sample, whereupon 10 to 30 g. shall be taken for analysis.

(*b*) Asphalt mastics or mastic cake shall be warmed on a hot plate or in a hot oven until soft enought to be broken up or stirred so that a representative sample for analysis may be taken. The amount

[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

taken for analysis will depend upon the amount of coarse gravel or stone in the mixture. The larger the gravel or stone, the larger will be the sample required for accuracy. The size of samples to be taken shall be as follows: Where all particles pass a 10-mesh sieve, 10 to 30 g.; where 25 per cent of the aggregate is retained on a 10-mesh sieve, 50 g.; where 50 per cent of the aggregate is retanied on a 10-mesh sieve, 100 g.; and where 75 per cent of the aggregate is retained on a 10-mesh sieve, 200 g.

NOTE.—Where the properties of the extracted bituminous matter are to be determined, 500 g. shall be taken and extracted with pure benzol as described in Sections 4 and 6 and examined as described in Section 7.

III. APPARATUS.

(*A*) *For Analysis of 10 to 30-g. Samples.*

Apparatus, 10 to 30-g. Samples.

3. In cases where a 10 to 30-g. sample is sufficient, the analysis shall be carried out by means of the glass extractor described in Section 8 of the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

(*B*) *For Analysis of 50 to 500-g. Samples.*

Apparatus, 50 to 500-g. Samples.

4. The apparatus for analysis of samples containing coarser aggregate shall be the large extractor shown in Fig. 1, consisting of a large brass cylinder, through the bottom of which projects a 16-candle-power incandescent carbon filament bulb to supply heat to the extraction apparatus proper, which is held in the upper portion of the cylinder. This apparatus is composed of a cylindrical brass vessel for holding the solvent, a cylindrical wire basket made of 80-mesh wire cloth suspended in the cylinder, and an inverted conical condenser which serves as a top.

IV. PROCEDURE.

(*A*) *Analysis of 10 to 30-g. Samples.*[2]

Procedure.

5. An ordinary Whatman or S. and S. Filter Thimble shall be dried and weighed. The weighed sample shall be placed in the thimble and 40 to 50 cc. of CS_2 poured over the sample. The thimble containing the sample shall be suspended under the condenser by a fine wire bail. The flask shall be cautiously heated by a steam-bath or electric heater just enough to vaporize the solvent. Cold water is circulated through the condenser. The heat evaporates the carbon disulfide in the flask. This condenses upon the condenser and drops back upon

[1] 1924 Book of A.S.T.M. Standards.

[2] This method and the method for analysis of larger samples are especially adapted for asphalt mastics, grouts and mastic cake. If the binder is coal-tar pitch, the "free carbon" constituent of the binder will remain with the mineral aggregate. The committee has under consideration a possible separate method for analysis of coal-tar mastics.

the sample through which it filters, thus dissolving out the bitumen which collects in the bottom of the flask.

The extraction should be discontinued when the CS_2 drops colorless from the filter. The time of extraction will depend upon the nature of the bitumen and mineral aggregate in the sample and upon the degree of heat applied, the coldness of the water in the condenser and other factors. In some cases extraction may be complete in one hour, in others four or five hours may be necessary.

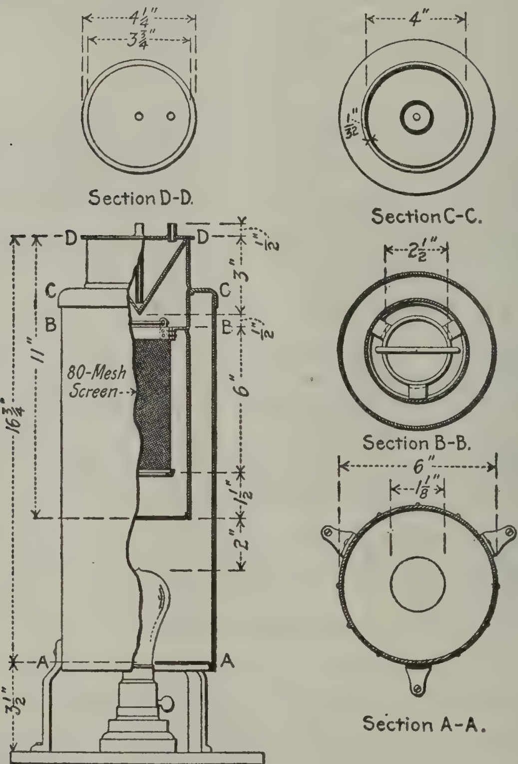

FIG. 1.

When the solvent comes through clear, the filter shall be removed and washed with a fine jet of CS_2 from a washing bottle to wash out any bitumen that may be retained at the top of the paper and to break up any channels that may have been formed by the CS_2 passing through. If the washings show any color, the thimble shall be put back and extraction continued until the solvent again becomes colorless. It shall then be removed, dried carefully, at a low temperature at first to

prevent ignition of the absorbed CS_2, and finally to constant weight at 100° C. (212° F.), cooled and weighed.

The solution in the flask shall be rinsed into a weighed porcelain or silica evaporating dish or crucible and the solvent burned off under a hood. The residue shall be ignited over a flame or in a muffle and the ash weighed and the weight added to that of the mineral matter in the filter paper. This is to correct for the fine mineral matter which will be carried through the paper by the solvent. Should there be a considerable amount of ash recovered in this way, and if it is found that the mineral matter is calcium or other carbonate, it shall be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat. Ordinarily, however, the mineral matter going through the paper will be so small in amount that the difference caused by ignition may be neglected.

The corrected loss in weight on the original sample represents the percentage of bitumen.

The mesh analysis of the mineral aggregate shall be made in accordance with the Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, except Fine Aggregates Used in Cement Concrete (Serial Designation: D 7) of the American Society for Testing Materials.[1] The extracted residue shall now be transferred from the thimble to the 200-mesh sieve, the paper being gently rubbed to free adhering particles.

The aggregate on the sieve shall be gently rubbed with the fingers to break up lumps and to free any particles of fine dust that might adhere to larger sand particles. The sieve shall be shaken over a piece of paper from side to side with the right hand, striking it sharply against the palm of the left hand until no appreciable amount of dust comes through the sieve on to the paper. The paper shall be cleared from time to time by raising one side with the left hand and rolling off the siftings, so that it can be seen when the sifting is complete.

The material remaining on the 200-mesh sieve shall be weighed and the amount of 200-mesh material which has passed through shall be determined by difference.

This operation shall be repeated upon the coarser sieves in order and the amount passing each sieve and retained on the next finer recorded as percentage of the original sample.

(*B*) *Analysis of 50 to 500-g. Samples.*

6. A large filter paper, 12 or 13 in. in diameter, shall be fitted inside the wire basket of the extractor by folding once more than in

[1] 1924 Book of A.S.T.M. Standards.

ordinary filtering, or by wrapping it over a form which fits inside the basket (a cylindrical bottle of proper size makes a good form) and placing it inside the basket.

The basket with contained filter paper shall be dried and weighed. The sample shall be weighed and packed in the filter paper in the basket. Care should be taken not to pack all coarse particles in one place and the fine particles in another but to have them mixed together in uniform proportions.

The sample shall be covered with a disk of felt or wad of absorbent cotton to insure even distribution of the dropping solvent, thus preventing it from forming a channel through the sample. The basket shall be suspended in the extractor and 150 to 200 cc. of CS_2 poured over the felt or cotton. The condenser shall be placed over the top and water circulated through it. Current shall be started through the electric lamp underneath the extractor and the extraction carried on exactly as in the smaller glass extractor, but on a larger scale. The time for extraction will vary from 3 to 12 or more hours, depending upon the nature of the sample.

To determine when extraction is complete, the condenser shall be raised and the basket lifted out to observe if drippings are clear. One or two drops caught upon white filter paper should leave but a light stain.

The drying and weighing of the basket, burning off of the solution for correction, and the sifting of the mineral aggregate shall be carried on exactly as in the case of the smaller sample as described in Section 5, except that in sifting mineral aggregates containing coarse stone, it is well to remove the stone by putting the mineral aggregate first through a 10-mesh sieve, as the large stone would injure the 200-mesh sieve.

The stone removed by the 10-mesh sieve may be screened through $\frac{1}{4}$-in., $\frac{1}{2}$-in., and coarser screens in order, as described in the Standard Method of Mechanical Analysis of Broken Stone or Broken Slag, except Aggregates used in Cement Concrete (Serial Designation: D 18) of the American Society for Testing Materials[1] and the fine material passing the 10-mesh sieve through 200-mesh and coarser sieves in order, all amounts passing any one screen or sieve and retained on the next finer one being reported as percentage of the original sample.

Thus the ingredients of the mixture may be roughly separated by the use of the proper sized sieves, and examined for physical and chemical characteristics.

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[1] 1924 Book of A.S.T.M. Standards.

NOTE.—The siftings passing the 200-mesh sieve will consist largely of the dust or pulverized rock used in the mixture. The material between the 100-mesh and 10-mesh will consist largely of the sand used in the mixture, with any particles of fine crushed stone within these limits that existed in the original materials. Gravel or broken stone as a rule will be larger than 10 or 8 mesh.

Carbon tetrachloride, benzol, or chloroform may be used instead of carbon disulfide, with the only difference that in the case of non-inflammable solvents, the solvent will have to be evaporated from the solution of bitumen to determine the ash for correction instead of burning off directly. When the solvent is expelled, the bitumen can be ignited for ash.

(C) Recovery and Examination of Extracted Bitumen.

7. The benzol solution containing the asphalt shall be evaporated to a thick viscous liquid on a steam or water bath, then transferred to a steam oven at 105° C. (221° F.) until its weight is nearly equal to the percentage of asphalt calculated in accordance with Section 5. It shall then be transferred to a hot plate and heated carefully with stirring at a temperature not exceeding 121° C. (250° F.) until its weight is exactly that determined in accordance with Section 6.

(D) Solubility of Aggregate in Hydrochloric Acid.

8. Where the mastic is to be used for acid-proof floors, tank linings, etc., pulverized silica and other materials insoluble in acid are used in preparing the mixture. The most important chemical property, therefore, is solubility or insolubility in mineral acids. This determination may be carried out in the following manner:

About 2.5 g. of the dust, or of the coarser material, or stone which has been previously pulverized in a mortar shall be dried to constant weight at 325° F. The loss will represent any moisture that may have been retained in the material as well as any absorbed solvent that may not have been expelled while drying the sample after extraction.

A 1-g. sample of the dried material shall be placed in a 250-cc. beaker, covered with a watch glass and 10 cc. of concentrated c. p. hydrochloric acid added, diluted with 50 cc. of distilled water. Any lumps shall be broken up by means of a glass stirring rod. Any effervescence which may occur shall be noted. If considerable effervescing takes place, the sample may be said to be carbonate. If all the sample, or practically all, dissolves, the determination need not be carried farther. If apparently insoluble, it shall be heated carefully, brought to boiling for 15 minutes and filtered through a tared Gooch filter, washing the insoluble residue with hot water. It shall be ignited and weighed and the loss reported as the percentage soluble in hydrochloric acid.

TENTATIVE METHODS OF TESTING SMOOTH-SURFACED ASPHALT ROLL-ROOFING, SLATE-SURFACED ASPHALT ROLL-ROOFING AND SLATE-SURFACED ASPHALT SHINGLES[1]

Serial Designation: D 228 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

Scope. 1. These methods cover the examination of roofings and shingles composed of roofing-felt, saturated and coated on both sides with asphalt, and surfaced on the top with either powdered or granulated mineral matter, and on the under side with powdered mineral matter.

I. SAMPLING

Sampling. 2. From each shipment or portion thereof representing a product of the same kind, class and weight, a number of rolls or bundles shall be selected at random, equivalent to one-half the cube root of the total number of rolls or bundles included in the lot, except that in lots of 1000 or less, five rolls or bundles shall be taken. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the nearest higher whole number. For convenience, the following table is given, showing the number of samples to be selected from shipments of various sizes.

Packages in Shipment	Number of Packages Selected	Packages in Shipment	Number of Packages Selected
Up to 1000	5	5 833 to 8 000	10
1001 to 1728	6	8 001 to 10 648	11
1729 to 2744	7	10 649 to 13 824	12
2745 to 4096	8	13 825 to 17 576	13
4097 to 5832	9	17 577 to 21 952	14

II. DETERMINATION OF SIZE AND WEIGHT

(*A*) *Smooth-roll and Slate-roll Roofing*

3. Each roll selected shall be stripped. The wrappers, packing material, nails and cement taken from each roll shall be weighed

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 441 Lexington Ave., New York City.

together to the nearest $\frac{1}{4}$ lb. and the maximum weight thereof per roll shall be recorded. The quantity and character of nails and cement shall be recorded.

4. Each roll shall be unwound, discarding any detached surfacing that may fall off in so doing, and the workmanship and finish of the roofing observed.

5. The length and width of each roll shall be measured to the nearest $\frac{1}{4}$ in., and the square feet of material contained in each roll calculated.

6. The rolls shall be rewound, fastened with pieces of light string and then each roll shall be weighed to the nearest $\frac{1}{4}$ lb. The weight of roofing per 108 sq. ft. contained in each roll shall be calculated and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the entire shipment.

7. The average weight per 108 sq. ft. for the rolls examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the entire shipment.

8. From the rolls examined, the one whose weight is nearest the average weight of the lot shall be selected. The first convolution or two shall be unrolled, and with a knife and straight edge, the sheet shall be cut across at right angles to the edges. A section measuring exactly 30 in. in the direction of the roll's length shall then be removed. The width of this section shall be measured to the nearest $\frac{1}{32}$ in. and weighed with an accuracy of $\frac{1}{4}$ oz. The weight in pounds per 108 sq. ft. shall then be calculated from the following formula:

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of 30-in. section in ounces}}{\text{Width of 30-in. section in inches}} \times 32.4$$

This must check within 1 per cent of the average weight per 108 sq. ft. of the shipment (Section 7). If this sample fails to do this, then additional samples shall be cut from the same roll, until one is obtained which does, which sample shall be kept for further examination.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, a 30-in. sample shall be taken and examined separately from each roll sampled in Section 2.

(*B*) *Slate-surfaced Shingles*

9. Each bundle selected shall be stripped. The packing material shall be weighed to the nearest $\frac{1}{4}$ lb., recording the weight separately for each bundle. The maximum weight of packing material per "square" shall be recorded.

10. The shingles in each bundle, *a*, shall be counted, discarding

any detached surfacing that may fall off in so doing. The workmanmanship and finish of the shingles shall be observed.

11. The shingles contained in each bundle shall be weighed to the nearest $\frac{1}{4}$ lb. (b). The dimensions of the shingles shall be accurately measured and the net area of material in each shingle calculated in square feet (c).

12. The area in square feet of material contained in each bundle ($a \times c$) shall be calculated. The weight of roofing material per 108 sq. ft. contained in each bundle $\left(\frac{b}{a \times c} \times 108\right)$ shall be calculated, and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the entire shipment.

13. The average weight (d) per 108 sq. ft. for the bundles examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the entire shipment. Also, the average weight per shingle $\left(\frac{c \times d}{108}\right)$ shall be calculated.

14. From the bundles selected, a definite number of representative shingles, whose weight shall fall within 1 per cent of the average weight ascertained in Section 13 shall be taken for further consideration. The number of shingles so selected shall represent as closely as possible 6 sq. ft. of material, and shall be selected as far as possible from different bundles.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, then a similar sample shall be taken and examined separately from each bundle of shingles sampled in Section 2.

III. DETERMINATION OF PLIABILITY OF SMOOTH-ROLL ROOFING AND SLATE-ROLL ROOFING

15. With a photographic trimmer five strips shall be cut from the sample of roofing, each 1 in. in width and 6 in. in length, in the direction of the fiber grain, and immersed in water at 77° F. (25° C.) for 10 to 15 minutes. These strips shall be removed from the water and immediately bent through 180 deg. at uniform speed, in exactly 2 seconds, over a mandril of the following size:

Smooth-roll Roofing—At least 3 of the 5 strips shall not crack over a mandril 10 mm. in diameter.

Slate-roll Roofing—At least 3 of the 5 strips shall not crack on a mandril 25 mm. in diameter.

IV. DETERMINATION OF BEHAVIOR ON HEATING

16. Two strips shall be cut from the sample of roofing or shingles, each approximately 8 by 8 in. They shall be weighed and suspended

vertically in the center of an air-oven maintained at 176° F. (80° C.) ± 5°F. (3° C.) with a thin wire fastened through holes punctured near one edge. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The strips shall be maintained at the prescribed temperature for exactly two hours, then cooled, removed carefully and each specimen weighed. The average loss shall be calculated as a percentage. Any change in appearance of the specimen shall be recorded, such as flowing, sagging, blistering or absorption of the surface coatings; also sliding of granular surfacing.

V. DETERMINATION OF SATURANT IN MOISTURE-FREE FELT

17. Two strips shall be cut from the sample of roofing or shingles, each approximately 2 in. in width and 8 in. in length. They shall be warmed in an oven at a temperature of approximately 176° F. (80° C.). Then with a knife, the front and back coatings shall be peeled off, care being taken to remove as little as possible of the saturated felt, and to assure that all of the coatings and surfacing are stripped from the felt. The saturated felt so obtained shall be weighed. It shall be extracted with benzol, chloroform, carbon tetrachloride or carbon disulfide, in a suitable extractor and the extraction continued until the drippings have become colorless. The desaturated felt shall be removed from the extractor, placed in a tared vessel, heated in a ventilated oven at 225° F. (107° C.) for 30 minutes, cooled in a desiccator, and the felt finally weighed as rapidly as possible. The heating shall be repeated until the weight of the felt remains constant as determined by two consecutive weighings taken not less than 10 minutes apart, which shall show a further loss of not more than 0.1 per cent. From the weight so obtained and the original weight, the percentage of saturation carried by the moisture-free felt shall be computed.

VI. DETERMINATION OF WEIGHT OF MOISTURE-FREE FELT

18. Two strips shall be cut from the sample of roofing or shingles, each approximately 2 in. in width and 8 in. in length. Each of these shall be accurately weighed in grams and the length and width measured to within 1 mm. or $\frac{1}{32}$ in. It shall be extracted with benzol, chloroform, carbon tetrachloride or carbon disulfide, in a suitable extractor and the extraction continued until the drippings have become colorless. The desaturated felt shall be removed from the extractor, being careful not to lose any adhering mineral matter, placed in a tared vessel, heated in a ventilated oven at 225° F. (107° C.) for 30 minutes,

cooled in a desiccator, any adhering surfacing material brushed off and retained and the felt finally weighed as rapidly as possible. The heating shall be repeated until the weight of the felt remains constant as determined by two consecutive weighings taken not less than 10 minutes apart, which shall show a further loss of not more than 0.1 per cent. The benzol extract and accompanying mineral matter shall be retained for further examination. From the weights so obtained and the respective areas of the strips of desaturated felt, the weight of moisture-free felt shall be computed in pounds per 108 sq. ft., from the following formulas and the results averaged:

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of specimen in square centimeters}} \times 221.2$$

or

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of specimen in square inches}} \times 34.3$$

VII. DETERMINATION OF ASH IN MOISTURE-FREE FELT

19. The strips of desaturated felt obtained as described in Section 18 shall be cut into squares about 2 cm. on each side and thoroughly mixed. Pieces amounting to about 10 g. shall be selected at random and dried at 225° F. (107° C.) for 30 minutes. They shall be cooled in a desiccator, accurately weighed and then incinerated in a weighed crucible either over an open flame or in a muffle until all the carbon is consumed. A few drops of ammonium carbonate solution shall then be added, the sample ignited gently and weighed. The percentage of ash shall be calculated on the basis of the moisture-free felt.

VIII. DETERMINATION OF MINERAL SURFACING AND MINERAL MATTER IN COATINGS

20. The slate and other mineral matter recovered from the extraction process described in Section 18 shall be combined with the mineral matter removed from the surface of the desaturated felt and that resulting from the evaporation and ignition of the benzol extract. The mineral matter shall be dried in air free from the odor of benzol, then heated in an oven for 30 minutes at 225° F. (107° C.), and finally cooled in a desiccator and weighed. From the combined weight of the specimens taken in Section 18, the total "mineral surfacing and mineral matter in the coatings" shall be calculated in pounds per 108 sq. ft.

TENTATIVE METHODS OF TESTING INSULATING VARNISHES[1]

Serial Designation: D 115 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1922, 1923, 1924, 1925.

Material Covered.

1. These tests are intended for varnishes which are applied by brushing, dipping or spraying, and are primarily for the purpose of providing electrical insulation.

I. SPECIFIC GRAVITY.

Specific Gravity.

2. The specific gravity shall be measured with a pyknometer, Westphal balance or with a hydrometer so graduated that the specific gravity can be determined to 0.001. The temperature of the varnish shall be not less than 18° C. (64.4° F.) nor more than 22° C. (71.6° F.) and corrected to 20° C. (68° F.) by applying a correction of 0.0007 per 1° C. (0.0004 per 1° F.).

II. VISCOSITY.

Viscosity.

3. (*a*) The viscosity shall be determined with a Stormer or a MacMichael viscosimeter. For the Stormer instrument, it shall be stated in terms of the viscosity of distilled water determined with the same instrument and under the same conditions. For the MacMichael viscosimeter, the viscosity shall be stated in absolute units, the centipoise (cp.). The short-tube type of efflux viscosimeter usually employed for lubricating oils is not acceptable. The temperature of the varnish shall be 20° C. (68° F.).

(*b*) The MacMichael viscosimeter shall be calibrated as follows:

Obtain fair-sized samples (2 to 5 gal. or more) of several oils of suitable viscosities. These should be selected so that they fall in the ranges of viscosity in which measurements are likely to be made. This is to permit calibration of the instrument with an oil of the same order of viscosity as the products that are to be tested. A convenient series of samples for the testing of insulating varnish is:

[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

Mineral Oil having an absolute viscosity of approximately 20 centipoises at 20° C. (approximately 65 seconds, Saybolt Universal Viscosity, at 37.8° C.).

Mineral Oil having an absolute viscosity of approximately 40 centipoises at 20° C. (approximately 100 seconds, Saybolt Universal Viscosity, at 37.8° C.).

Mineral Oil having an absolute viscosity of approximately 170 centipoises at 20° C. (approximately 300 seconds, Saybolt Universal Viscosity, at 37.8° C.).

Castor Oil having an absolute viscosity of approximately 992 centipoises at 20° C. (approximately 110 seconds, Saybolt Universal Viscosity, at 98.9° C.).

Retain the major portion of each of these samples and send 4 to 8 oz. to the Bureau of Standards, Washington, D. C., for determination of absolute viscosity. Specify 20° C. as the temperature at which measurements are to be made. The samples, if they are properly refined filtered oils, are regarded as stable for at least one year if kept uncontaminated in closed opaque containers.

NOTE.—Standardized samples of known absolute viscosity may be obtained directly from the Bureau.

(*c*) When using the MacMichael viscosimeter it is necessary (1) to obtain the zero adjustment prior to placing the varnish in the cup, (2) to adjust to a sufficiently low speed to prevent eddy currents.

(*d*) The report shall include such details as the kind of instrument used, the size of the counterweight if a Stormer instrument is used, or the size of the wire in case the MacMichael instrument is used, etc.

III. FLASH POINT.

Procedure for Flash Point.

4. The flash point shall be determined in accordance with the Standard Method of Test for Flash Point of Volatile Flammable Liquids (Serial Designation: D 56) of the American Society for Testing Materials.[1]

IV. TIME OF DRYING.

Test Specimen.

5. (*a*) Specimens for this test shall be pieces of thoroughly cleaned, smooth sheet copper or brass about 3 cm. (1.18 in.) wide and 20 cm. (7.88 in.) long and about 0.127 mm. (0.005 in.) thick.

(*b*) The specimen shall be dipped once in the varnish at a room temperature of approximately 20° C. (68° F.) and withdrawn slowly and uniformly (about 38 cm. (15 in.) per minute). The consistency of the varnish shall be first so adjusted by trial that, when dry as determined in accordance with Section 7, the thickness of the film of varnish on each side of the metal shall be between 0.022 mm. (0.0009 in.) and 0.026 mm. (0.001 in.). Care shall be taken before dipping the specimens that the varnish has stood in the dipping tank for a sufficient length of time to be free from air bubbles.

[1] 1924 Book of A.S.T.M. Standards.

Procedure.

6. (*a*) Specimens of air-drying varnish shall be dried in dust-free air at a room temperature of approximately 20° C. (68° F.).

(*b*) In the case of baking varnishes, six specimens shall be dipped and allowed to drain at a room temperature of approximately 20° C. (68° F.) until the varnish is set as indicated when the impression left on the surface by pressing lightly thereon with a finger will not become obliterated by further flow of the material. They are then to be dried in dust-free air in an oven at 105 to 110° C. (221 to 230° F.). At the end of the first 30 minutes, and again at the end of each 10-minute period thereafter, one specimen shall be taken from the oven and examined. In the case of slow-drying varnishes, this 10-minute period may be lengthened at the discretion of the operator.

Time of Drying.

7. The varnish shall be considered dry when a specimen will not stick to itself when folded and pressed together between the thumb and finger at a temperature of approximately 20° C. (68° F.).

V. DIELECTRIC STRENGTH TEST.

Dielectric Strength Test Specimen.

8. (*a*) Specimens for the dielectric strength test shall be prepared by dipping pieces of thoroughly cleaned, smooth sheet copper or brass about 20 cm. (7.88 in.) square and about 0.127 mm. (0.005 in.) thick into the varnish which shall be at the consistency prescribed in Section 5 (*b*).

(*b*) Each specimen shall be dipped twice, as specified in Section 5 (*b*), once in each direction, in order to give a more uniform thickness of coating. The specimen shall be dried after each dip in the same vertical position in which it was dipped.

(*c*) Specimens of air-drying varnish shall be dried in dust-free air after each dip at a room temperature of approximately 20° C. (68° F.) for a period 600 per cent longer than that determined in accordance with Section 7, provided such period does not exceed 24 hours.

(*d*) Specimens of baking varnish shall be drained and then baked in dust-free air after each dip for a period 300 per cent greater than that determined in accordance with Section 7 provided such period does not exceed 24 hours.

(*e*) The final thickness of the film of varnish on each side of the specimen shall be between 0.044 mm. (0.0018 in.) and 0.052 mm. (0.002 in.).

Procedure.

9. (*a*) The dielectric strength of the two films of varnish shall be determined by applying alternating potential to two circular metal disks, 5.08 cm. (2.0 in.) in diameter and with edges rounded to a radius of 0.64 cm. (0.25 in.) which are placed in contact with the two sides of the specimen directly opposite each other and under a pressure

of approximately 0.5 kg. (1.1 lb.). Starting at zero, the voltage shall be increased uniformly to breakdown at a rate of 0.5 kilovolt per second, except that if breakdown occurs at this rate in less than 40 seconds, the rate shall be decreased so that breakdown will occur in not less than 40 seconds. If the material fails at less than 5 kilovolts, the minimum time shall be reduced from 40 seconds to 20 seconds. Ten such punctures are to be made at various points selected at random on each specimen. In each test the thickness of the films of varnish is to be determined as close to the point of puncture as practicable.

NOTE.—When necessary, in order to get ten punctures, additional specimens should be tested.

(*b*) The frequency of the test potential shall be not greater than 100 cycles per second, and each part of the testing apparatus shall have a continuous rating of not less than 2 kva. (preferably larger). The wave form shall be a sine curve as defined, and the voltage shall be measured by methods approved by the American Institute of Electrical Engineers.[1]

(*c*) The voltage may be controlled by any approved method which does not distort the wave form beyond the limits prescribed above and which does not subdivide the voltage in steps greater than 500 volts. The apparatus shall comply with the Standards of the American Institute of Electrical Engineers.

Dielectric Strength. 10. The volts at puncture, the net thickness of insulation and the volts per mil of net thickness shall be reported for each of the ten tests together with the average maximum and minimum volts per mil.

VI. WATER ABSORPTION TEST.

Procedure. 11. Specimens similar to those described in Section 8 shall be immersed in water at a room temperature of approximately 20° C. (68° F.) for a period of 24 hours. Upon removal from the water, the surface water shall be wiped off and dielectric strength tests made immediately as described in Section 9.

Dielectric Strength. 12. The volts at puncture, the net thickness of the insulation and the volts per mil of net thickness shall be reported for each of the ten tests, together with the average, maximum and minimum volts per mil.

[1] Standards of the American Institute of Electrical Engineers.

VII. HEAT ENDURANCE TEST.

13. For the heat endurance test, specimens shall be prepared as in Section 8. After removing not less than 1.27 cm. (0.5 in.) from one edge of the specimens, the number of strips required by Section 14 (*a*) shall be cut from the same edge, each 1.9 cm. (0.75 in.) in width. **Test Specimens.**

14. (*a*) After setting as show by the test indicated in Section 6 (*b*), the strips referred to in Section 13 shall be placed in a uniformly heated oven in which the temperature is maintained at 100° C. (212° F.) within ± 5° C. (9° F.). A strip shall be removed at the end of 1, 2, 4, 8 and 24 hours respectively and every 24 hours thereafter. These, together with the initial strip, shall be tested as follows at a room temperature of approximately 20° C. (68° F.). **Procedure.**

(*b*) Each strip shall be bent through 180 deg. over a rod 0.32 cm. ($\frac{1}{8}$ in.) in diameter. The number of hours of baking at which first cracking in the insulation occurs shall be noted and reported.

VIII. ACID AND ALKALI PROOF TEST.

15. The specimens to be used for the test for acid and alkali proofness shall be brass rods 1.5 cm. (0.59 in.) in diameter, 15 cm. (5.90 in.) long and carefully rounded at one end to a radius of 0.75 cm. (0.295 in.). These specimens shall be dipped three times into the varnish, leaving exposed about 3 cm. (1.18 in.) of the rod at the end opposite the rounded end. Each coat shall be dried 25 per cent longer than the period determined in Section 7. Three specimens each shall be prepared for the acid and alkali solutions. **Test Specimens.**

16. (*a*) The three specimens shall be suspended in the acid or alkali whose effect it is desired to determine to within 3 cm. (1.18 in.) of the end of the coated portion of the rod and suitable provision made for detecting the change in the electrical resistance between the rod and the solution. (Note 1.) **Procedure.**

(*b*) It is recommended that these tests be made in 10-per-cent solutions as follows:

Sulfuric acid of sp. gr. 1.069 at 60° F. (15.5° C.) or nitric acid of sp. gr. 1.056 at 60° F. (15.5°) C.) or hydrochloric acid of sp. gr. 1.050 at 60° F. (15.5° C.) and sodium hydroxide of sp. gr. 1.115 at 60° F. (15.5° C.).

(*c*) The temperature of the solution shall be kept at approximately 20° C. (68° F.).

NOTE 1. A simple method is to connect a voltmeter between each rod in turn and one side of a 110-volt direct current circuit, the other side of the circuit being connected to the solution through any piece of suitable metal suspended in the solu-

tion. The resistance will be inversely proportional to the deflection of the voltmeter pointer, that is, the smaller the deflection, the greater the resistance. Failure of the material will, therefore, be indicated by a sudden increase in the deflection of the voltmeter pointer.

Acid and Alkali Proofness. 17. The resistance between each rod and the solution shall be measured once per day and the number of days elapsing before breakdown occurs shall be taken as the "proofness" of the varnish.

IX. OIL PROOF TEST.

Oil Test Specimen. 18. For the test for the effect of oil, pieces cut from the specimens prepared for the dielectric strength test (Section 8) may be used after they have been punctured and measured.

Procedure. 19. The effect of oil on the varnish shall be determined by immersing the specimens in transformer oil at a temperature of 100° C. (212° F.) for 48 hours and noting the effect on the varnish as indicated, for example, by wiping with a piece of dry white cloth.

Note.—Incipient disintegration of the surface of the varnish may sometimes be detected by examining the oil for turbidity. If a specimen of the oil filtered through filter paper can be distinguished from an unfiltered sample when the two samples are held in front of a strong light, the oil is turbid.

X. DRAINING TEST.

(Also known as "Working Viscosity" Test)

Test Specimen. 20. A strip of bond paper 0.064 mm. (2.5 mils) in thickness, 10.2 cm. (4 in.) in width and 50.8 cm. (20 in.) in length, shall be immersed in the varnish at a room temperature of approximately 20° C. (68° F.) up to a line previously drawn across the paper a few inches from the top. The paper shall be withdrawn at a slow and uniform rate (about 38 cm. (15 in.) per minute), care being taken that the varnish is free from air bubbles. The specimen shall be permitted to drain thoroughly at room temperature while suspended in a vertical position. It shall then be dried or baked (according to the type of the varnish) until dry as determined in accordance with Section 7.

Measurements. 21. The thickness of the specimen in mils shall be measured at points 5.1 cm. (2 in.), 17.8 cm. (7 in.) and 30.5 cm. (12 in.), respectively, from the line to which the specimen was immersed.

Report. 22. The thickness of each film in mils at the three points specified in Section 21 shall be recorded. The difference between the thickness at the upper point (5.1 cm.) and that at the lower point (30.5 cm.) shall be taken as a measure of the variation in the film thickness caused by draining.

XI. EVAPORATION TEST.

23. One hundred cubic centimeters of the varnish shall be placed in a flat-bottom crystallizing dish approximately 75 mm. (2.95 in.) in diameter and 45 mm. (1.77 in.) in height. It shall be heated to a temperature of 100° F. (37.8° C.) ± 2° F. (1.1° C.) for a period of 7 hours, the sample being exposed to still air in the open room. **Procedure.**

24. The decrease in volume of the sample shall be taken as the evaporation, this decrease being determined by noting the amount of water or kerosene that must be added to fill the dish to the original level. **Evaporation.**

NOTE.—This test is relative only. That is, it is only suitable for comparing one varnish with another when the tests are made simultaneously under exactly the same conditions.

XII. TEST FOR NON-VOLATILE MATTER.

25. A portion of the sample shall be placed in a stoppered bottle or weighing pipette and weighed. About 1.5 g. of the sample shall be transferred to a weighed flat-bottom metal dish about 8 cm. (3.15 in.) in diameter, such as the cover of a friction-top tin can. The container shall again be weighed and the exact weight of the portion of the sample transferred to the weighed dish calculated by difference. The dish with its contents shall be heated for three hours in an oven maintained at 105 to 110° C. It shall then be weighed after cooling. **Procedure.**

26. The ratio of the weight of the residue to that of the original sample expressed as a percentage shall be taken as the percentage of non-volatile matter in the varnish. **Non-Volatile Matter.**

TENTATIVE METHODS OF TESTING ELECTRICAL PORCELAIN[1]

Serial Designation: D 116 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1924.

Material Covered. 1. These tests are intended to apply to porcelain which is to be used for electrical insulating purposes.

I. TENSILE STRENGTH.

Apparatus. 2. Any standard testing machine may be employed, but it shall be of suitable capacity.

Preparation of Test Specimens. 3. Test specimens may be prepared by pugging or extruding rolls of suitable size, throwing rolls by hand or by cutting rolls from pugged blocks. For comparative tests one method of preparing specimens should be adhered to, as test specimens prepared by different processes show different results. They shall be turned in the green condition to such dimensions as will give fired pieces of standard size, shrinkage being determined and allowed for as in regular ceramic practice. Variation from the standard dimension shall be kept within $\pm$ 5 per cent, as it has been found that the strength per square inch calculated from results obtained with test specimens of about 0.2 sq. in. increases about 0.5 per cent for a decrease in area of 1 per cent.

Test specimens shall be fired in saggars on end, either on placing sand or clay bats. They shall not be buried in sand as this will cause the pieces to break in firing. Firing shall be carried out in the regular porcelain kilns. Specimens which have warped in firing shall be rejected because they will give unreliable results and therefore a sufficient number of specimens should be prepared to give the required number of satisfactory ones.

Specimens. 4. (*a*) *Ohio Brass Co. Method.*—The test specimens shall have the shape and dimensions shown in Fig. 1.

[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

(*b*) *Jeffry-Dewitt Insulator Co. Method.*—The test specimens shall have the shape and dimensions shown in either Fig. 2 or Fig. 3. The maximum cross-section area in the reduced section of the specimen shall be not greater than 0.2 sq. in.

When dry, the shoulder of the specimens shall be glazed, leaving the reduced section free from glaze.

NOTE.—The glaze is necessary to obtain uniform results and insure that failure occurs in testing in the reduced section. The glaze is omitted from the reduced section in order to avoid variation in strength which may be attributable to the glaze.

Mounting of Specimens.

5. (*a*) *O.B. Method.*—The method of mounting the test specimen in the specimen holders is shown in Fig. 4. The method of using the assembly jig shown in the figure is as follows: The test specimen is calipered at the small section. The tapered jig, *G*, with the arbor, *E*, is then placed in the guides, *V*. The point on the tapered piece, *G*, which has the same diameter as the minimum section on the

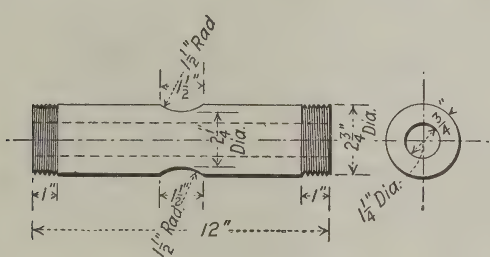

FIG. 1

specimen is placed directly over the V-notched block, *N*. This piece is then raised until it comes in contact with the piece, *G*, and the wing nut, *S*, tightened. *E* and *G* are then removed and the cap pieces, *C*, screwed on to the rods, *R*, which are laid in the notches *V-V*. The center of the specimen is placed in the V-notched block, *N*, and the clamping piece, *L*, tightened down to hold it in place.

One of the caps, *C*, with the attached rod, *R*, can then be moved over the end of the specimen and held in place by tightening the strap, *H;* the whole assembly apparatus is then stood on one end and the cap moved so as to allow filling with cement. The cap is then moved up in place, rotating it at the same time so as to properly work in the cement. The strap, *H*, is then tightened and the piece left undisturbed until the cement hardens.

When the cap has been attached at one end, the assembly apparatus can be stood on the other end and the other cap cemented in place.

Note.—This arrangement permits of setting up the jug properly and easily for specimens which vary slightly in diameter and insures keeping the center line of the specimen in the center line of the caps. A slight tip of the specimen will be unimportant so long as both caps can be assembled to the specimen.

After the cement has hardened (which will not require more than five or ten minutes if litharge cement is used), the specimen can be removed and eyes screwed into caps in place of the rods, *R*.

(*b*) *J-D Method.*—The method of mounting the test specimen in this method of testing is as follows:

Test Specimen, Fig. 2 (Conical Ends).—The specimen shall be held by small steel clamps, Fig. 5, each consisting of a split bushing, *B*, ground to fit the test specimen, *T*, and held by a collar, *C*, the load being applied by the plate, P_1, held in slots of the jaws of the testing machine through the ball and socket joint, *J*. The soft gasket, *G*,

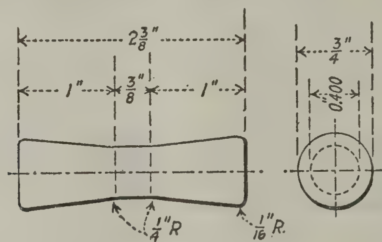

Fig. 2.

of blotting paper shall be inserted between the porcelain and the bushing to distribute the load. A new gasket shall be used with each specimen.

Test Specimen, Fig. 3 (Dumb-bell Ends).—The specimen is held by a split ring, *R*, Fig. 6, with the same radius of curvature as the shoulders of the specimen. This ring fits into a recess in plate P_2 which, in turn, fits into the slots in the jaws of the testing machine.

Note.—No gasket is required with this piece when the shoulders are glazed. Satisfactory results have not been obtained with unglazed pieces, even when using a gasket. This form of specimen has the advantage of requiring no gasket and so is more rapid in manipulation and of allowing greater variation in dimensions.

Method. 6. (*a*) Not less than 10 specimens (preferably more) shall be tested in the normal condition.

(*b*) The specimen shall be pulled apart at such a constant speed that the beam of the testing machine can be kept well balanced at all loads.

(*c*) When failure occurs outside of the reduced section, that result shall be discarded.

(*d*) The tests shall be made at a room temperature of about 20° C. (68° F.).

7. The report shall include the following: **Report.**

(*a*) The diameter in inches or centimeters as measured by a micrometer at the point of fracture.

(*b*) The breaking load of each specimen in pounds or in kilograms.

(*c*) The ultimate strength in pounds per square inch or in kilograms per square centimeter of each specimen as calculated from the actual area of the specimen measured at the point of fracture.

(*d*) The average of the results given in Paragraph (*c*).

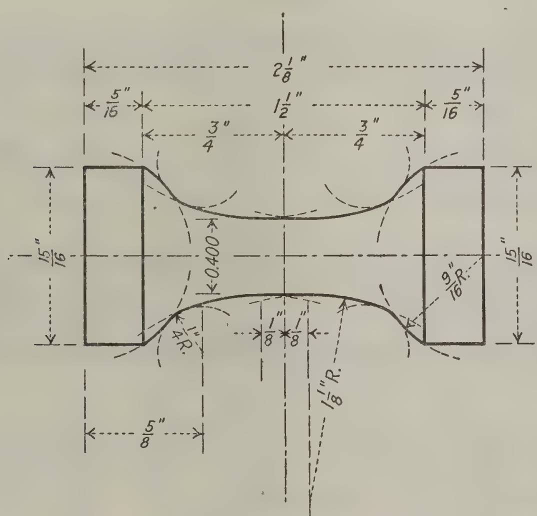

FIG. 3.

(*e*) The character of the material tested and a description of its behavior under stress.

(*f*) The speed in inches or centimeters per minute at which the jaws traveled during the test.

II. COMPRESSIVE STRENGTH.

8. Any standard testing machine may be used. A contact pad or cushion of $\frac{1}{32}$ in. of blotting paper shall be placed between the upper and lower faces of the specimens and the heads of the testing machine to equalize irregularities in the surfaces. Fresh cushions shall be used for each specimen tested. **Apparatus.**

9. The test specimen shown in Fig. 7 shall be used for the compressive strength test. **Specimens**

Method. 10. (*a*) Not less than 10 specimens (preferably more) shall be tested in the condition in which they are received.

(*b*) The load shall be applied at that constant rate of speed which will permit the beam to be kept well balanced at all loads.

(*c*) The test shall be made at a room temperature of about 20° C. (68° F.).

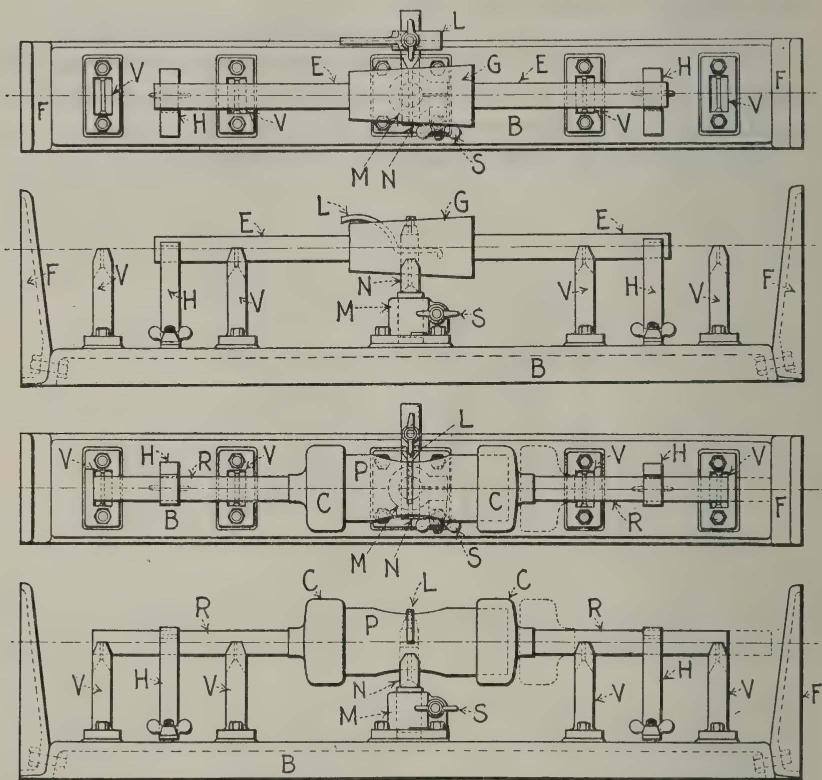

FIG. 4.

Report. 11. The report shall include the following:

(*a*) The dimensions of each specimen in inches or in millimeters.

(*b*) The load in pounds or kilograms on each specimen at the first sign of fracture.

(*c*) The ultimate compressive strength in pounds per square inch or in kilograms per square centimeter for each specimen calculated from the measured area before the load is applied.

(*d*) The average of the results given in Paragraph (*c*).

(*e*) General character of the material tested and a description of its behavior under load.

(*f*) The speed in inches or in centimeters at which the jaws traveled during the test.

III. RESISTANCE TO IMPACT.

12. The test shall be made with an apparatus in which a weight of 1 lb. is raised $\frac{1}{2}$ in. and allowed to fall on the specimen. It is then raised 1 in. and allowed to fall again on the specimen. This is continued, the weight being raised and dropped through a distance increasing by increments of $\frac{1}{2}$ in. until the specimen is broken. **Apparatus.**

13. The test specimen shown in Fig. 7 shall be used for impact tests. **Specimen.**

14. (*a*) The test specimen shall be clamped in the base of the impact tester directly under the hammer. The stops shall be set to **Procedure.**

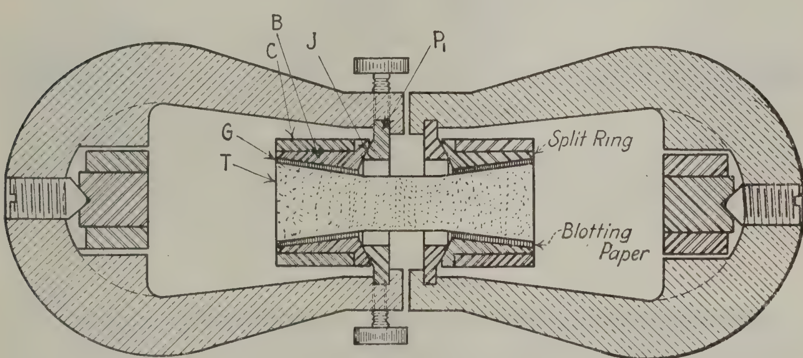

FIG. 5.

give a successively increasing rise of hammer by $\frac{1}{2}$-in. increments, the first height being $\frac{1}{2}$ in.

(*b*) The entire hammer shall weigh 1 lb.

(*c*) The test shall be continued until the specimen cracks.

(*d*) Not less than five specimens shall be tested in the condition in which they are received.

15. The report shall include the number of blows required to break each specimen, the energy of each blow in inch-pounds and the total energy in inch-pounds (that is, the sum of all blows). **Report.**

NOTE.—The apparatus may be a simple hand-operated one constructed along the line of the one shown in Fig. 8 or a motor-operated machine where the weight is lifted and the distances are increased automatically.

IV. DIELECTRIC STRENGTH.

Apparatus. 16. The apparatus shall be as described in Section 14 of the Standard Methods of Testing Molded Insulating Materials (Serial Designation: D 48) of the American Society for Testing Materials.[1]

Specimens. 17. The specimens shall be as shown in Fig. 1 except that the threads may be omitted.

Method. 18. (*a*) *Porcelain for all purposes.*—A pad of wet clay about $\frac{1}{4}$ in. thick shall be laid in the groove and a lining of wet clay about $\frac{1}{4}$ in. thick put on the inside of the specimen. The potential shall be applied to these two pads of clay by any suitable means. Mercury may be substituted for the wet clay if desired. The test should preferably be made in air but it may be made in transformer oil if necessary.

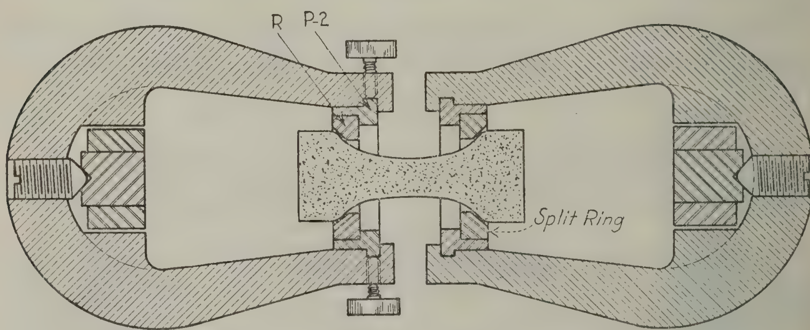

FIG. 6.

Not less than ten specimens shall be punctured in their normal condition at a normal room temperature of about 20° C. (68° F.).

(*b*) *Porcelain for transmission line insulators.*—In addition to the test prescribed in Paragraph (*a*), not less than 10 specimens shall be tested in the same manner at a temperature of 75° C. (167° F.).

(*c*) *Porcelain for spark plugs.*—In addition to the test prescribed in Paragraph (*a*), not less than 10 specimens shall be tested to determine the effect of high temperatures on the dielectric strength. The specimens shall be placed in an electrically heated oven the temperature of which can be raised at a constant rate to 600° C. (1112° F.). A given voltage shall be applied and maintained constant and the temperature raised at any constant rate such that the porcelain is always practically at the temperature of the air. The temperature shall be raised until puncture occurs.

(*d*) Whenever a puncture occurs at a point other than the mini-

[1] 1924 Book of A.S.T.M. Standards.

mum section of the specimen in any of the tests prescribed above, that result shall be discarded.

19. The report shall include the following: Report.

(*a*) A statement of the purpose for which the porcelain is intended and the kind of tests which were made.

(*b*) The thickness of the specimen at the bottom of the groove.

(*c*) The voltage at puncture for each of the test specimens together with the average, maximum and mimimum volts per mil or per millimeter of thickness.

V. RESISTANCE TO THERMAL CHANGE.

(*A*) *Porcelain for Transmission Line Insulators.*

20. The apparatus shall consist of a hot water bath maintained at a temperature of 100° C. (212° F.) and an ice water bath maintained at 0° C. (32° F.). Apparatus.

21. The test specimens shall be as shown in Fig. 1. Specimens

22. (*a*) The test specimen shall be immersed in the ice water bath for ten minutes and then transferred as quickly as possible to the hot water bath, and allowed to remain there for ten minutes. The specimen shall be transferred back to the cold water and the cycles continued until the specimen breaks. Method.

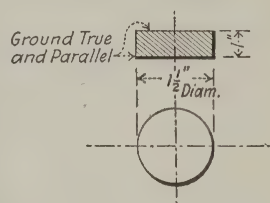

FIG. 7.

(*b*) Not less than five specimens shall be tested.

23. Report the number of cycles necessary to cause fracture of each test specimen. Report.

(*B*) *Porcelain for Spark Plugs and Heating Devices.*

24. A furnace in which a temperature of 900° C. (1652° F.) can be obtained and any suitable testing machine for determining the transverse strength of small beams may be used. Apparatus.

25. The specimens shall be as shown in Fig. 3. Specimens.

26. (*a*) One-half of not less than 12 specimens shall be taken and placed in the furnace. The rate of heating shall be so adjusted that the temperature reaches 900° C. (1652° F.) in two hours. The furnace shall then be allowed to cool at such a rate that room temperature is reached in four hours. Method.

(*b*) All specimens for transverse strength shall be tested by placing them on supports 6 in. apart and loading them at the center.

27. The report shall include the following: Report.

(a) The load in pounds or kilograms required to break each specimen not subjected to heat treatment, together with the average.

(b) The load required to break each specimen that was subjected to heat treatment, together with the average.

(c) The percentage loss of strength due to heat treatment calculated from the two averages.

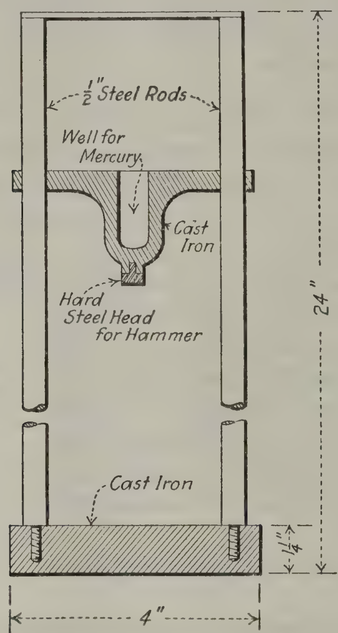

FIG. 8.

VI. POROSITY.

(A) *Water Absorption.*

Apparatus. 28. Any good chemical balance, a beaker of distilled water at normal room temperature of about 20° C. (68° F.) and an oven of any standard make capable of maintaining a uniform temperature at the desired point within ± 5° C. shall be provided.

Specimens. 29. A single piece of porcelain shall be used weighing from 30 to 50 g. and with at least 50 per cent of the surface newly fractured.

Method. 30. The specimen shall be dried for 24 hours at 120° C. (248° F.), cooled in a desiccator and weighed. The specimen shall then be totally submerged in the distilled water at room temperature (about 20° C.) and allowed to remain submerged for 100 hours. The water shall be boiled for approximately one hour during the first, twenty-

fifth, forty-ninth and seventy-third hours. The specimen shall be removed at the end of the 100-hour period, the surface moisture carefully dried off with a clean, dry cloth and the specimen weighed.

31. The report shall include the following: **Report.**

(*a*) The original weight of the specimen;

(*b*) The dry weight of the specimen;

(*c*) The dry weight of the specimen after immersion for 100 hours;

(*d*) The percentage of moisture content in each specimen as received and the percentage of moisture absorbed during 100 hours, taking the dry weight as 100 per cent, and the average where more than one specimen is tested.

(*B*) *Penetration of Dye.*

32. A closed receptacle containing an alcoholic dye solution and a pump with which a pressure up to 600 lb. per sq. in. can be maintained in the receptable shall be provided. **Apparatus.**

33. A single specimen of porcelain shall be used, weighing at least 30 g. and with at least 50 per cent of the surface newly fractured. This specimen may be taken from any piece of finished ware, but if the piece varies materially in thickness (as, for example, in a transmission line insulator), a specimen shall be taken from both the thinnest and the thickest portions. **Specimen.**

34. The specimen shall be immersed for two hours in a saturated methol alcoholic solution of eosin or fuchsine at a pressure of not less than 200 lb. per sq. in. nor more than 600 lb. per sq. in. at a room temperature of about 20° C. (68° F.). **Method.**

35. The report shall include a statement of the maximum depth of penetration of the dye toward the interior of the specimen from the fractured surface exposed to the solution. (Lines of penetration along obvious cracks caused by possible fracturing are to be disregarded.) **Report.**

TENTATIVE METHODS OF TESTING SHEET AND TAPE INSULATING MATERIALS FOR DIELECTRIC STRENGTH[1]

Serial Designation: D 149 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1922; Revised, 1923, 1924.

Scope. 1. These methods are designed to determine the dielectric strength of sheet and tape insulating materials. Since the dielectric strength of such materials is dependent upon the time of application of the electrical tension, two tests differing in time duration are included as *A*, Short-Time Dielectric Strength Test, and *B*, One-Minute Step-by-Step Dielectric Strength Test, respectively.

A third test, designated as an Endurance Dielectric Strength Test, is included under *C*. It shows the relative dielectric strength of different flexible sheet and tape insulating materials at high temperature when subjected to electrical tensions for relatively long periods.

It should be noted that the results obtained with the electrodes specified for sheet material will differ from those specified for tape material (Section 3) and will not be comparable because the apparent dielectric strength of thin materials varies with the area of the electrodes.

A. Short-Time Dielectric Strength Test

I. APPARATUS

High-Voltage Transformer. 2. (*a*) Any well-designed, high-tension transformer connected to an alternating current supply, having as nearly a true sine wave as possible, may be used. The transformer and the source of supply of energy shall be not less than 2 kva. for voltages of 50,000 volts or less, and not less than 5 kva. for voltages above 50,000 volts. The frequency shall not exceed 100 cycles per second.

(*b*) Regulation shall be so controlled that the high-tension testing voltage taken from the secondary of the testing transformer can be

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

raised gradually from any point and in no case more than 500 volts at a step. The control may be made by generator field regulation, with an induction regulator, or with a variable ratio auto transformer. Any method of regulating the voltage is satisfactory which does not distort the wave more than 10 per cent from a sinusoidal shape.

(*c*) The voltage may be measured by any approved method which gives root-mean-square values, preferably by means of a voltmeter connected to a special voltmeter coil in the high-tension winding of the testing transformer, or to a separate step-down instrument potential transformer. A voltmeter on the low-tension side of the transformer is satisfactory, if the ratio of transformation does not change under any test condition. An electrostatic voltmeter properly calibrated in the high-tension circuit is also satisfactory. A spark gap may be used to check the readings at very high potentials.

(*d*) Some protection is desirable in the high-tension circuit of testing transformers where the potential is 25,000 volts or over, to prevent dangerous surges and limit the current when the test specimen is punctured. It is, however, desirable to have as much energy available as possible when puncture occurs. If impedance in the form of choke coils is used in series with the high-tension terminals, it should not be greater than that which will limit the high-tension current to double the normal rated current of the testing transformer.

When a spark gap is used, a non-inductive resistance of about one ohm per volt should be inserted in series with one terminal of the spark gap, to damp high-frequency oscillations at the time of breakdown and limit the current flow. This resistance shall be as near the gap as possible. If the test is made with one side grounded, this resistance shall be on the ungrounded side of the circuit, and if neither side is grounded, the resistance shall be inserted one-half on each side of the spark gap. Water tube resistors are preferable to carbon for this purpose, as carbon resistance may be materially decreased by the passage of current.

(*e*) The apparatus used and the method of measuring the voltage shall meet the requirements of the Standards of the American Institute of Electrical Engineers.

Electrodes.

3. The electrodes shall be of brass or copper with flat polished contact surfaces. For sheet material they shall be cylinders 2 in. (50.8 mm.) in diameter and 1 in. (25.4 mm.) in length with the edges rounded to a radius of $\frac{1}{4}$ in. (6.35 mm.). For tapes the electrodes shall be cylindrical rods $\frac{1}{4}$ in. (6.35 mm.) in diameter with edges rounded to a radius of $\frac{1}{32}$ in. (0.8 mm.) and the upper movable one shall weigh 0.1 lb. (45.4 g.) $\pm$ 0.005 lb. (2.3 g.).

II. TEST SPECIMENS

Test Specimens.

4. (*a*) The test specimens may be of any convenient size, but the total area of the specimens of one sample of material shall be sufficient to permit making at least ten satisfactory tests.

(*b*) The specimens shall be representative of the material to be tested, care being taken to select material which is free from abnormal defects such as blisters, wrinkles, cracks, etc.

III. PROCEDURE

Surrounding Medium.

5. The tests shall be made in air with the specimen at room temperature.

Condition of Electrodes.

6. The testing electrodes shall be kept clean and polished.

Position of Electrodes and Specimen.

7. The electrodes shall be self-aligning, and shall be placed exactly opposite one another with the specimen in a horizontal plane between them.

8. The edges of tapes shall be clamped between blocks of insulating material under a pressure of approximately 100 lb. per sq. in. to prevent flashover occurring before puncture.

NOTE.—In the Appendix are shown two different devices which have been found satisfactory for these tests.

Application of Voltage.

9. Starting at zero, the voltage shall be increased uniformly to breakdown at a rate of 0.5 kilovolt per second, except that if breakdown occurs at this rate in less than 40 seconds, the rate shall be decreased so that breakdown will occur in not less than 40 seconds. If the material fails at less than 5 kilovolts, the minimum time shall be reduced from 40 seconds to 20 seconds.

Number of Tests.

10. Ten tests shall be made and the average of these ten puncturing voltages shall be taken.

Thickness.

11. The thickness of the specimen shall be measured at each puncture, using a ratchet micrometer graduated to 0.001 in. and having a circular foot not less than 0.240 in. nor more than 0.260 in. in diameter.

Report.

12. The report shall include the following:

(*a*) The thickness of the specimen at each puncture;

(*b*) Total volts and volts per mil of thickness at each puncture;

(*c*) The average, maximum and minimum volts at puncture per mil of thickness for each sample;

(*d*) The room temperature;

(*e*) The relative humidity, in per cent;

(*f*) The duration of the test.

B. One-Minute Step-by-Step Dielectric Strength Test

IV. APPARATUS

13. The apparatus shall consist of the high-voltage transformer and the electrodes specified in Sections 2 and 3. **Apparatus.**

V. TEST SPECIMENS

14. The test specimens shall be as specified in Section 4. **Test Specimens.**

VI. PROCEDURE

15. The tests shall be made in air with the specimen at room temperature. **Surrounding Medium.**

16. The testing electrodes shall be kept clean and polished. **Condition of Electrodes.**

17. The electrodes shall be self-aligning, and shall be placed exactly opposite one another with the specimen in a horizontal plane between them. **Position of Electrodes and Specimen.**

18. The edges of tapes shall be clamped between blocks of insulating material under a pressure of approximately 100 lb. per sq. in. to prevent flashover occurring before puncture.

NOTE.—In the Appendix are shown two different devices which have been found satisfactory for these tests.

19. A voltage shall be applied which is equal to 40 per cent of the breakdown voltage obtained in the short-time test to the nearest even 1000 volts (or 500 volts with low voltages). The voltage shall then be increased by 10 per cent of the initial value until failure occurs, the voltage being held at each step for one minute. When changing from one step to a higher step, the voltage shall be increased to the required value within 10 seconds. **Application of Voltage.**

20. Five tests shall be made and the average of these five puncturing voltages shall be taken. **Number of Tests.**

21. The thickness of the specimen shall be measured at each puncture, using a ratchet micrometer graduated to 0.001 in. and having a circular foot not less than 0.240 in. nor more than 0.260 in. in diameter. **Thickness.**

22. The report shall include the following: **Report.**

(*a*) The thickness of the specimen at each puncture;

(*b*) Total volts and volts per mil of thickness at each puncture;

(*c*) The average, maximum and minimum volts at puncture per mil of thickness for each sample;

(*d*) The room temperature;

(*e*) The relative humidity, in per cent;

(*f*) The duration of the test;

(*g*) The value of the initially applied voltage and the value of the voltage at each step.

C. Endurance Dielectric Strength Test

VII. Apparatus

Apparatus. 23. The apparatus shall consist of the high-voltage transformer specified in Section 2.

Test Specimen. 24. (*a*) The specimens shall be made by wrapping the material, in tape form with one-quarter lap, one-half lap, butt jointed or as sheets, on brass tubes. The tubes shall be, for sheets and for tape up to $1\frac{1}{2}$ in. in width, about 36 in. in length and 1 in. in outside diameter. For tapes wider than $1\frac{1}{2}$ in., the tubes shall be 2 in. in outside diameter. The sample shall be built up to the desired thickness, simulating practical conditions, winding all layers of tape in the same direction.

(*b*) A smooth layer of metal foil shall then be wrapped over the insulation for a distance of 24 in., leaving 6 in. of insulation uncovered at each end of the tube. The metal foil shall be bound in place with a wrapping of adhesive tape and shall extend the full length of the tube in order to protect the insulation at the end from corona discharge.

(*c*) A thermocouple shall be applied to the metal foil near the center of the tube and held in place by the adhesive tape.

VIII. Procedure

Position of Specimen. 25. The specimen shall be mounted in an oven and a temperature of 100° C. (212° F.) maintained during the test. Provision shall be made for so mounting the specimen that the tube may be connected to the high voltage side of the circuit with the metal foil sheath and thermocouple connected to ground.

Application of Voltage. 26. A voltage equal to 10 per cent of the breakdown voltage (to the nearest kilovolt) obtained in the short-time test (Test *A*) shall be applied and maintained for 30 minutes. The voltage shall then be increased by steps of 20 per cent of the initial value until puncture occurs, the voltage being held at each step for 30 minutes.

Temperature Readings. 27. The temperature of the sample as indicated by the thermocouple shall be observed at intervals during the test and recorded at the end of each 30-minute period. It will be found that the temperature rises gradually until just before breakdown when the temperature increases rapidly. During this latter period, temperature readings shall be recorded at frequent intervals.

Report. 28. (*a*) The results shall be plotted using time, expressed in hours, as abscissas, and temperature, in degrees Centigrade, as ordinates. In addition to time as abscissas, the values of applied kilovolts shall be indicated since the value of kilovolts increases uniformly with

time. The significant values to be taken from the curve and reported are:

(1) Duration of test;
(2) Breakdown voltage;
(3) Temperature of specimen at breakdown;
(4) Rate of temperature rise during test.

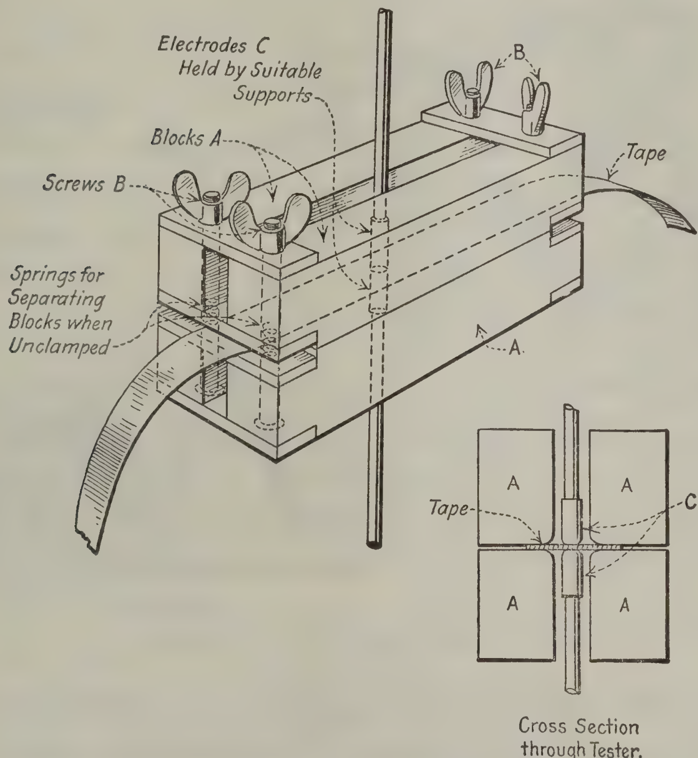

FIG. 1.—Elementary Form of Tape Tester.

(*b*) The report shall also include a description of the specimen and its preparation, the thickness of the insulation and the number of layers of insulation.

APPENDIX

Referring to Fig. 1, the four wooden clamping blocks, *A*, are held together by the screws *B*. These blocks are impregnated with paraffin which assists in sealing the joints between the wooden blocks and the tape. The electrodes are introduced into the space between the blocks at *C*. The tape can be advanced

through the blocks, for each shot, by opening and closing the clamping screws. In order to secure satisfactory results, the blocks must have accurately finished surfaces and should be carefully aligned.

Referring to Fig. 2, the two impregnated wooden clamping blocks, *A*, are pressed together by the spring, *B*, which is secured to the latched handle, *C*. Releasing the latch allows the blocks, *A*, to be separated to insert the tape or examine the electrodes or contact surfaces of the blocks.

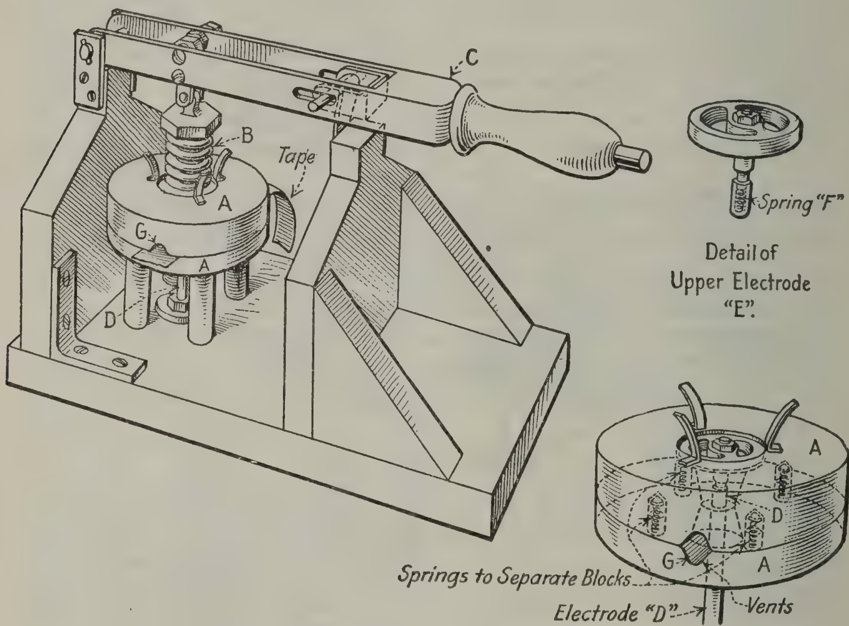

FIG. 2.—Lever-Operated Form of Tape Tester.

The lower electrode, *D*, is fixed in position with its top flush with the lower block *A*. The upper electrode, *E*, is removable and its contact face is pressed against the tape by the internal spring, *F*. The spring *B* seats on the electrode *E*, which rests on a shoulder in the block *A*.

The openings, *G*, provide the necessary vents for the arc which occurs when the tape under test fails. The successful operation of this device depends upon the accuracy of the contact faces of the blocks *A*, their alignment, the impregnation of the blocks *A* and the vent openings *G*.

TENTATIVE METHODS OF TESTING ELECTRICAL INSULATING MATERIALS FOR PHASE DIFFERENCE (POWER FACTOR) AND DIELECTRIC CONSTANT AT RADIO FREQUENCIES[1]

Serial Designation: D 150 – 23 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922; REVISED, 1923

1. This method of test is intended to apply to all solid electrical insulating materials. **Scope.**

The behavior of material under test at radio frequencies is fundamentally different from that at low frequencies.

In this test the phase difference of the dielectric is determined at a measured frequency by determining the effective resistance (or equivalent series resistance) and capacity of a condenser made up with the material as the dielectric. The dielectric constant is calculated from the measured capacity of such a condenser, and the thickness and area of the dielectric.

The dielectric power loss is proportional to the product of the dielectric constant and the phase difference.

I. APPARATUS.

2. The circuit in which the radio frequency waves are generated is called the generating circuit. The source of radio frequency voltage shall be an electron tube oscillator the frequency of which can be varied over the range desired and which can be coupled loosely to the measuring circuit. It shall furnish sufficient power so that the presence of the measuring circuit will not reduce its output. The output of the oscillator must be very constant, as any variation in voltage will cause a much larger variation in the final results, because of the differential method used. For this reason it is highly desirable to have the **Generating Circuit.**

[1] Criticisms of these Tentative Methods are solicited and should be directed, preferably before January 1, 1926, to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

plate current supplied by a storage battery or other source of very constant power.

Measuring Circuit. 3. The circuit in which the electrical insulating material specimen is placed for test is called the measuring circuit.

This circuit is composed of:

A condenser employing the test specimen as the dielectric and two metallic surfaces as the conducting plates.

A standard condenser of a special design having negligible phase difference or power factor.

One or more special inductance coils.

A low-resistance thermo-element (preferably less than five ohms).

A special double-pole double-throw switch equipped with mercury wells.

A set of resistor links having direct current resistance values ranging from negligible to about 40 ohms.

A sensitive low-resistance wall galvanometer of the deflecting mirror type or other suitable type of indicating instrument.

The arrangement of the apparatus in the measuring circuit is shown in Fig. 6 of the Appendix.

Frequency Meter. 4. The instrument used to measure the frequency of oscillation of the current in the generating circuit is called a frequency meter or wave meter. This instrument may be of any commercial design covering the wave-frequency range of about 85.7 to 1500 kilocycles per second (corresponding to wave lengths of 3500 to 200 meters) and capable of calibration accurate to within 1 per cent over the wave-frequency range used.

II. TEST SPECIMEN.

Test Specimen. 5. (*a*) For testing molded materials, the test specimen shall be a disk having the dimensions shown in Fig. 13 of the Appendix. A raised rim about the circumference is provided to hold a layer of mercury to form the upper plate when the specimen is used as a condenser.

(*b*) For testing laminated stiff sheet materials, either circular or rectangular test specimens may be used and the specimen shall be of such dimensions that, after fixing the upper mercury surface diameter (or length and width) at 2 cm. less than that of the specimen, the electrical capacity shall be not less than 100 micro-microfarads (100 $\mu\mu$f.). Specimens 25.4 by 30.4 cm. (10 by 12 in.) are recommended for general use.

(*c*) For testing solid waxes the test specimen may be cast to the shape mentioned in either Paragraph (*a*) or Paragraph (*b*).

(*d*) For testing very thin or flexible sheet material, such as varnished paper or mica, tin-foil may be used as the conducting condenser plates. The instructions for capacity and relative areas of conducting plates given in Paragraph (*b*) apply here. Where necessary, a mixture of beeswax and petrolatum may be employed as an adhesive for the tin-foil.

(*e*) For testing films of insulating varnishes, lacquers, paints, etc., the test specimen shall be as specified for dielectric strength tests in Sections 8 (*a*), (*b*), (*c*), and (*d*) of the Tentative Methods of Testing Insulating Varnishes (Serial Designation: D 115 – 25 T) of the American Society for Testing Materials,[1] except that the consistency of the varnish need not be adjusted. The copper or brass plate shall be one condenser plate and tin-foil the other. The tin-foil sheet shall not be larger than 18 cm. (7.09 in.) square. Both varnish films may be tested individually.

III. PROCEDURE.

(*A*) *Phase Difference* (*power factor*).

6. (*a*) The phase difference (power factor) of two specimens shall be measured by the resistance-variation method[2] in the condition received at normal room temperature of about 20° C. (68° F.) and at 5 radio-frequencies between 98 and 1200 kilocycles per second (3050 to 250 meters) each. **Method.**

(*b*) When practicable, the phase difference (power factor) of two specimens shall be measured by the same method and at radio frequencies within the same limits as mentioned in Paragraph (*a*) after the specimens have been immersed in water at a temperature of about 20° C. (68° F.) for 48 hours. The edges of the specimen shall have been protected from the action of the water by dipping it in melted paraffin to a depth of 10 mm. (0.394 in.). After removing the specimen from the water, the surface water shall be wiped off with a clean, absorbent cloth, being careful not to handle the paraffined edge, and then allowed to stand in the room away from drafts and direct sunlight for five minutes before making the measurements.

7. The specimen shall be floated on mercury in a glass dish on an insulating support levelled by means of levelling screws. Sufficient mercury shall be poured on the upper surface of the specimen to cover that surface within the confines of the barrier. In the case of molded materials, the confining barrier shall be of the material of the specimen; in the case of stiff sheet materials, the confining barrier shall be **Electrodes.**

[1] See p. 633.

[2] The principles of the resistance-variation method of measuring phase difference and dielectric constant are given in Bureau of Standards *Circular No. 74*, pp. 180–193.

a two-piece copper rectangle or ring made of 0.32-cm. ($\frac{1}{8}$-in.) square copper wire.

Procedure. 8. The details of the method of testing for phase difference are given in the Appendix.

Calculations. 9. The values of phase difference in degrees shall be calculated from the formula:

$$\text{Phase difference in degrees, } \Psi^\circ = 3.60\, RCf \times 10^{-7}$$

where f = frequency in kilocycles per second;
R = measured resistance of specimen condenser in ohms;
C = measured capacity of specimen in micromicrofarads;

or

$$\text{Phase difference in degrees, } \Psi^\circ = 0.1079\, \frac{RC}{\lambda}$$

where R and C are the same as above and λ = the wave length expressed in meters.

(B) *Dielectric Constant.*

Method. 10. The capacity of a condenser in which the specimen is the dielectric shall be determined by comparison with a standard variable air condenser under the conditions prescribed for phase difference measurements in Sections 6 (*a*) and 6 (*b*) with the specimen arranged as in Section 7.

Procedure. 11. The details of the method of determining capacity are given in the Appendix.

Calculations. 12. The dielectric constant shall be calculated from the formula:

$$\text{Dielectric constant } K = \frac{CT}{0.0885\, S}$$

where C = the capacitance of the specimen in micromicrofarads;
T = the average thickness of the dielectric expressed in centimeters;
S = the area of the upper metallic surface expressed in square centimeters;

or

$$K = \frac{CT}{0.224\, S}$$

where C = the capacitance of the specimen in micromicrofarads;
T = the average thickness of the dielectric expressed in inches;
S = the area of the upper metallic surface expressed in square inches.

IV. REPORT.

Report. 13. The report shall include the following:

(*a*) The description of the material: *i. e.*, filler or base, binder, grade, color and average thickness;

(*b*) Room temperature and relative humidity;

(*c*) A curve for each specimen, showing phase difference (power factor) and dielectric constant plotted as ordinate values, and wave frequencies (wave lengths) as abscissa values for the specimen as received and when practicable after immersion in water for 48 hours. Each curve sheet will, therefore, show two phase difference and two dielectric constant curves;

(*d*) The phase difference and dielectric constant of each specimen at wave frequencies of 100 and 1000 kilocycles per second (3000 and 300 meters) as read from the curves for each of the two conditions and the average phase difference and dielectric constant and their product at these same frequencies.

APPENDIX

DETAILS OF RESISTANCE-VARIATION METHOD OF MEASUREMENT OF PHASE DIFFERENCE (POWER FACTOR) AND DIELECTRIC CONSTANT

Introduction.—A test specimen for the measurement of phase difference and dielectric constant at radio frequencies consists of a piece of insulating material placed between metal plates, the whole constituting an electric condenser. It is quite convenient and satisfactory to use mercury or tin-foil as the metal for the condenser plates. The specimen of molded insulating material may be floated on mercury and a pool of mercury formed on top of the specimen, confined by the raised rim about the edge of the test specimen.

The phase difference of an electrical insulating material is

$$\psi = 90° - \theta$$

where θ is the phase angle between the current flowing and the voltage across a condenser which has the space between its conducting plates filled with the insulating material.

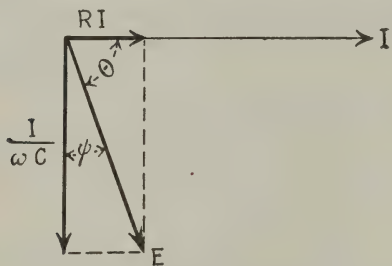

FIG. 1.—Vector Diagram.

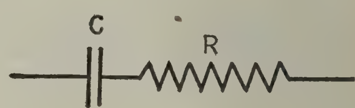

FIG. 2.—Condenser Circuit.

The voltage E (Fig. 1) across the condenser may be considered to be made up of a voltage $\frac{I}{\omega\ C}$ acting on the capacity and a voltage $R\ I$ acting on a resistance in series with the capacity. Thus, the condenser is, for the purpose of measurement, considered as equivalent to a pure capacity C with a resistance R in series with it (Fig. 2). This resistance is called "effective resistance" or "equivalent resistance" of the condenser, sometimes shortened to simply "resistance." This resistance is quite distinct from the direct-current or leakage resistance of the condenser.

The phase difference ψ may be calculated from the effective resistance and other quantities as follows: From Fig. 1.

$$\tan \psi = \frac{R\ I}{\frac{I}{\omega\ C}} = \mathrm{R}\ \omega\ C$$

where $\omega = 2\pi$ times the frequency of the current. Since for small angles $\tan \psi = \psi$, therefore

$$\psi = R\,\omega\,C.$$

The phase difference ψ is known if R, ω and C are known. The phase difference measurement thus involves a determination of three separate quantities. The formula $\psi = R\omega C$ is correct for ψ in radians, R in ohms and C in farads. With

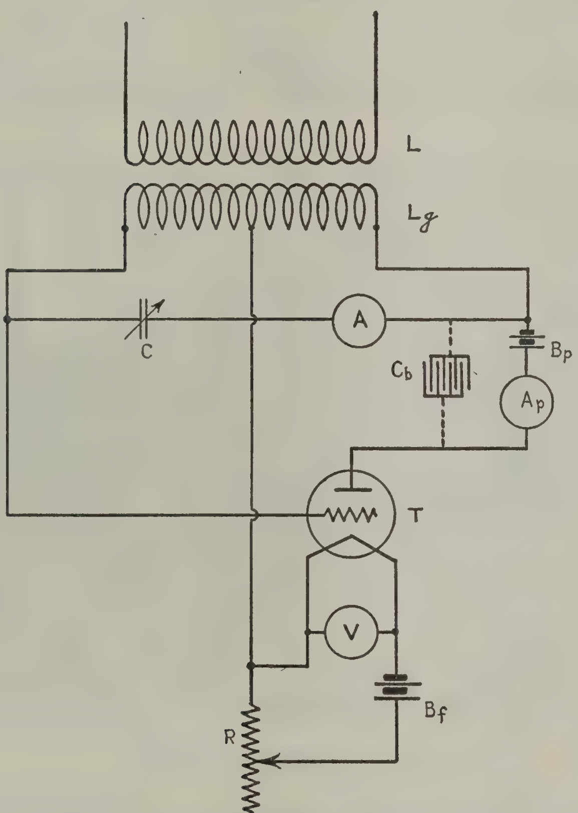

FIG. 3.—Radio-Frequency Generating Circuit.

the wave length in meters (λ_m) in the formula instead of ω, ψ in degrees instead of radians, R in ohms, and C in micromicrofarads instead of farads,

$$\psi^\circ = 0.1079\,\frac{R\,C}{\lambda_m}$$

The three quantities involved in the phase difference are measured with radio frequency currents. The current of a radio frequency is caused to flow in a circuit containing the condenser made up from the insulating material under test. The equivalent resistance (R) is determined by the resistance-variation method and the capacity is measured by comparison with a standard variable

air condenser. The determination of the equivalent resistance is the most important and difficult part of the measurement. The wave frequency or wave length (λ) is measured by a wave-meter placed near the generating circuit.

From the measurement of the capacity C of the test specimen, the value of the dielectric constant of the insulating material is easily obtained. The average thickness T of the dielectric and the area S of the upper mercury surface plate are measured. The dielectric constant is

$$K = \frac{C\ T}{0.0885\ S}$$

where C is expressed in micromicrofarads, T in centimeters and S in square centimeters.

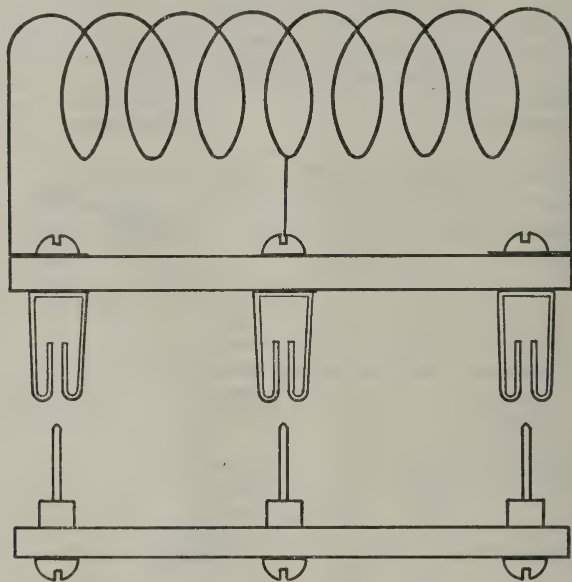

FIG. 4.—Secondary Inductance.

Circuits.—The measurement of phase difference and dielectric constant at radio frequencies requires a generating circuit and a suitable measuring circuit.

The generating circuit is shown schematically in Fig. 3. The inductance coil L_g is any one of a series of detachable tubular single layer inductors arranged somewhat as shown in Fig. 4. For the tests outlined in these specifications, it is probable that no more than five tubular inductors will be required. The smaller inductor may be made by winding 13 turns of wire on a 3½-in. tube with 6 turns on either side of the middle clip. The larger inductor may be made by winding 112 turns of wire on a 6¾-in. tube, 32 turns on either side of the middle clip and 24 turns outside of both outside clips. The other three inductors may be made to cover the gap between these two extremes.

The variable air condenser C may be of any suitable commercial shielded type having a maximum capacity of about 0.003 microfarads (3000 micro-

microfarads). The ammeter A is of the hot wire type having a range of 0 to 1 ampere. The by-pass condenser C_b may be any type of fixed condenser capable of withstanding 500 volts (direct current).

The plate battery B_p may be a 100 to 300-volt storage battery of the lead-acid type having an ampere capacity of about 0.2 ampere at the 8-hour discharge rate. Fig. 5 shows a 100-volt battery of this type with the supports fixed for stacking the batteries in an upright position. A lead couple and jar are also shown separately in this figure.

The ammeter A_p may be any type of direct-current instrument having a range of 0 to 50 milliamperes. The three-electrode electron tube T may be any of the so-called power tubes rated at 5 watts or over. The voltmeter V may be any type of direct-current instrument having a range suited to the type

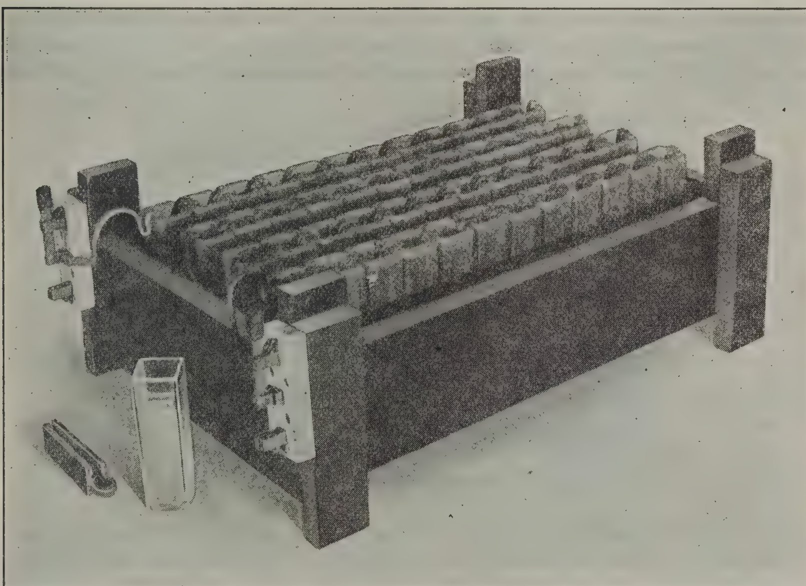

FIG. 5.—Storage Battery.

of electron tube used. The filament battery B_f should be of the lead-acid type whose voltage and ampere-hour capacity will be determined by the type of electron tube selected. The ampere carrying capacity and the resistance of the rheostat R would also be dependent on the type of tube selected.

The measuring circuit is shown schematically in Fig. 6. The standard variable air condenser C_s may be of the Bureau of Standards standard type having a maximum capacity of not to exceed 0.001 microfarads (1000 micro-microfarads). Fig. 7 shows the general construction of this type of condenser. This condenser is described more fully in *Circular No. 74* of the U. S. Bureau of Standards, "Radio Instruments and Measurements," Section 32. The special switch with mercury wells, together with a non-inductive resistor are shown in Fig. 8. The special switch is constructed throughout of insulating material

except the leads and wells which are of copper. The two upright standards of the switch are Pyrex glass rods about 1 cm. in diameter, while the supports which hold the six copper wells and leads are of Pyrex glass tubing. The copper wells are swaged to the copper leads and no solder used. The non-inductive resistor is made from a piece of glass tubing, two corks, two pieces of about No. 14 copper wire bent to form an *L* and a piece of small-diameter high-resistance wire. Several of these resistors are required, but in all cases the distance between the spurs should be the same, because the resistors must fit into the mercury wells, as shown in the switch (Fig. 8). Each of these units has a different value of resistance made so by varying either the length or the diameter of the resistance wire or both.

The condenser C_x is made either by floating the test specimen on mercury and pouring mercury on the upper surface until it is filled to the confining barriers or by the use of two sheets of tin-foil. The mercury bath on which the specimen rests is contained in a glass dish which rests on a piece of insulating material which in turn is supported by some sort of a levelling device.

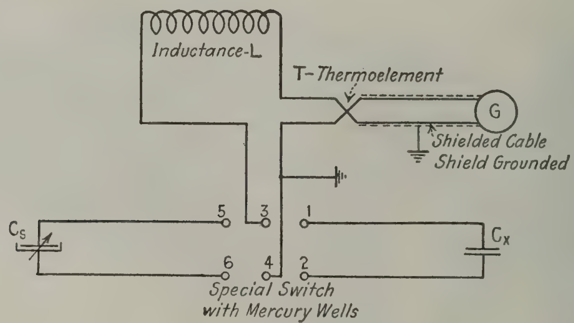

Fig. 6.—Measuring Circuit.

Fig. 9 shows the method of floating the specimen except that in this case a rectangular specimen is shown without a flange of the same material. A two-piece rectangular copper mercury retainer is used on the upper surface.

The standard inductor *L*, Fig. 6, may be of the type shown in Fig. 10, except that only standard binding posts will be required for connections. This inductor must be of some design having a comparatively low resistance so that the coil resistance will not be a large proportion of the combined equivalent resistance of the circuit made up of inductor *L* and the specimen condenser C_x. To cover the wave frequencies (wave lengths) named in these specifications, it would be desirable to have at least five inductors having inductance values of about 0.1, 0.5, 1, 2, and 3 millihenries.

The thermo-element *T* is of the crossed-wire type shown in Fig. 11. For this purpose the thermo-element base might better be equipped with four binding posts than with two binding posts and two clips. A cylindrical cap fits into the circular groove cut in the base and is held in place by a cap nut which screws on to the small brass bolt shown attached to the base. The wires used on this type of thermo-element may be of steel and "constantan" or "advance" about 0.02 mm. in diameter (see Bureau of Standards Radio

Laboratory Report cR251-2a). The resistance of the thermo-element should be from 3 to 5 ohms. A shielded cable connecting the thermo-element *T* to the galvanometer *G* may be standard lead-sheathed duplex cable.

The galvanometer *G* is a sensitive low-resistance wall galvanometer of the deflecting mirror type whose deflections within reasonable limits are proportional to the square of the current. A Leeds & Northrup No. 2285 high-sensitivity galvanometer with an external damping resistance of about 5 ohms, period about 2 seconds and concave mirror for about one meter focal distance has been found satisfactory.

Fig. 12 shows the complete apparatus as assembled in the Radio Laboratory of the Bureau of Standards. For more exact measurements the complete apparatus itself is housed in a small section of the room and completely surrounded with a fine mesh wire such as window screening. The purpose of this shielding is to insure the accuracy of measurement by shielding out the influence of outside radio-frequency generators. This view shows the wavemeter mounted on a tall stand, a rather complicated radio-frequency generator, a rack of 100-

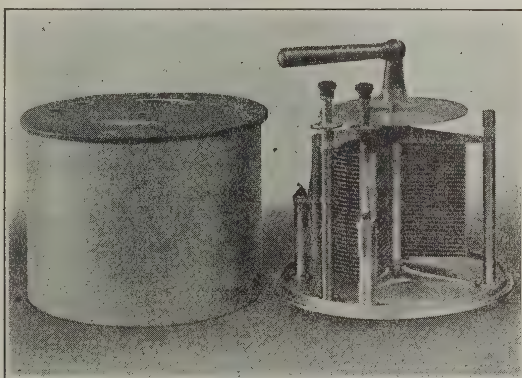

FIG. 7.—Standard Variable Air Condenser.

volt storage batteries, the bottle of distilled water for use with the batteries, the galvanometer rack showing the scale, lamp and galvanometer, the test specimen floated ready for measurement, the special skeleton switch, the standard inductor, the standard condenser and a box of non-inductive resistors. In addition, there will be observed a series of parallel wires between the generating circuit and the measuring circuit and also between the measuring circuit and the place occupied by the operator. The parallel wires between the generating circuit and the measuring circuit are all soldered together at the end nearest the operator (the left-hand end as shown), and left separate at the other end. The wire which connects all the parallel screening wires is then attached to a ground connection. The parallel wires between the measuring circuit and the operator are unattached at either end but all attached in the middle. This middle connector is also attached to the ground connection. The upper half of the shield between the operator and the measuring circuit is hinged to make it more easy to change the apparatus. It will be noticed that two panels of the over-all wire-netting screen have been removed in the photograph. The third

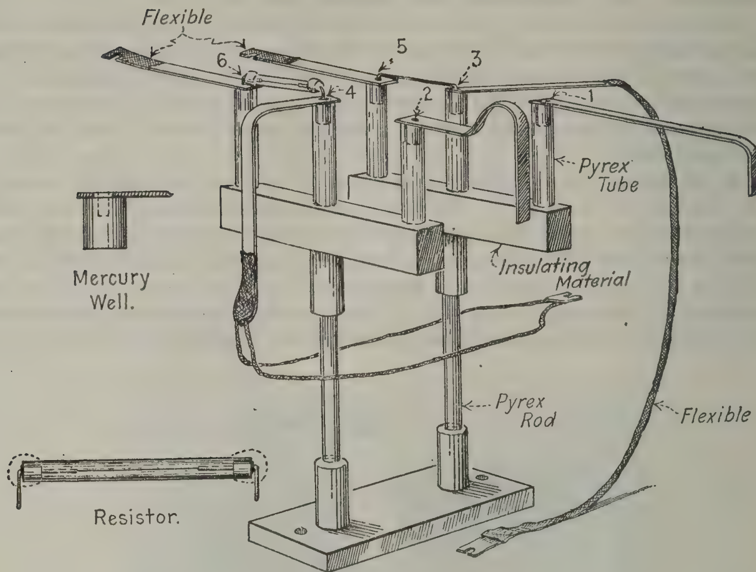

FIG. 8.—Special Switch.

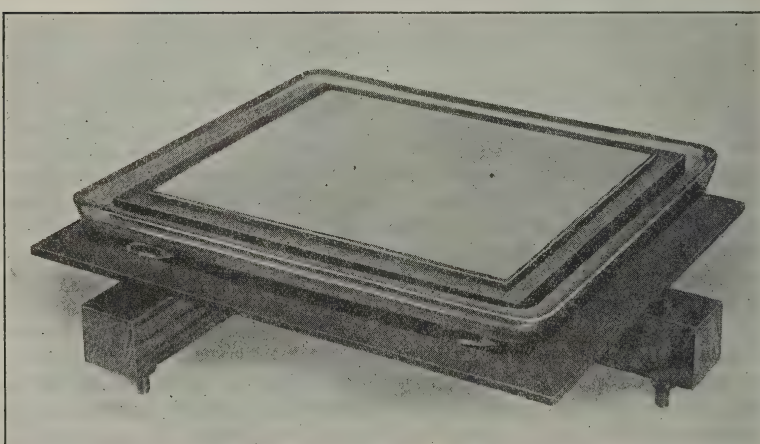

FIG. 9.—Test Specimen Floating on Mercury.

panel at the extreme right front of the photograph is hinged to form a door. This door permits the operator to arrange the test specimen in the mercury bath.

Method of Measurement.—Assume that the 13-turn inductor L_g is in position as shown in Fig. 3 and the other parts of the generating circuit are in readiness and generating radio-frequency current. Bring the generating circuit near to the measuring circuit with the shield of parallel wires between them as shown in Fig. 12. Put the 0.05-millihenry standard inductor L (Fig. 6) in position and insert heavy copper links between wells 1 and 3 and between 2 and 4. Write the value of the inductance L in Space No. 1 of the data sheet, Table I. Tune the generating circuit to the measuring circuit by varying the setting of the condenser C (Fig. 3), resonance being determined by the maximum deflection

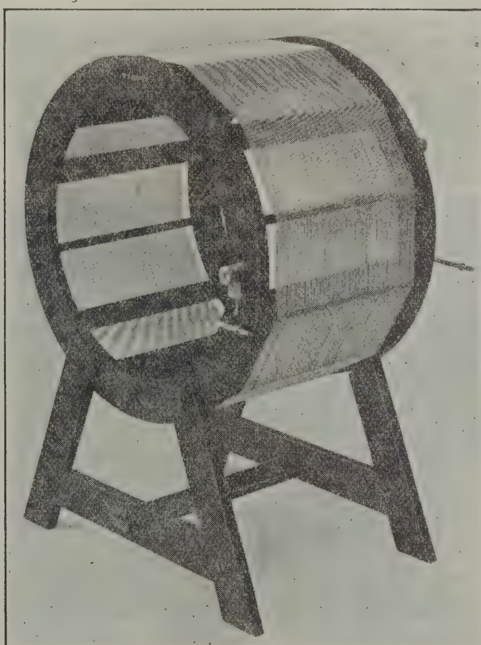

FIG. 10.—Standard Inductor.

of the galvanometer G (Fig. 6). It is essential that this tuning be done carefully. Change the coupling between L_g and L until the maximum deflection at resonance is about 40 cm. if a 50-cm. scale is used. Leave the generating circuit untouched and determine the wave frequency (wave length) emitted by the generating circuit by means of a frequency meter (wave meter). Record the wave-meter setting in Space No. 2, Table I. Remove the wave meter. Remove the copper link on the grounded side (that is between wells 2 and 4) and read the zero error of the galvanometer and record the value in Column No. 1 of the data sheet. Reinsert the copper link and record the maximum deflection in Column No. 2. Record the resistance of the heavy copper link as 0 in Column No. 5. Remove the copper link spanning wells 2 and 4 and read the zero deflection of

the galvanometer. Insert this as the second entry in Column No. 1. Pick out a non-inductive resistor of such resistance that it will cut the galvanometer deflection to about half the maximum value and insert between wells 2 and 4 to replace the copper link. Record the galvanometer deflection as a second

TABLE I.—SAMPLE RECORD OF TEST.

Date of Test,..........Specimen No.,..........Test No.,..........
Specimen Submitted by,..........Number Submitted,..........Date,..........
Base or Filler,..........Binder,..........Grade,..........Color,..........
Dry Bulb,.......° C.......° F. Wet Bulb,.......° C.......° F. Relative Humidity,.......%
Notes..........

Average thickness (T)..........(*No. 11*) cm. (*No. 11*) in.
Standard Inductance, (*No. 1*) mh.
Setting of { Wavemeter, (*No. 2*) deg. / Stand. Cond. No., (*No. 4*), (*No. 3*) deg. }
Upper plate area (S)..........(*No. 12*) cm.^{2} (*No. 12*) in.^{2}
Wave Freq., *kc*/sec., (*No. 5*) f.
Wave Length, (*No. 6*) Meters.
Specimen Capacity, (*No. 7*) $\mu\mu f$.
Phase Difference, $\Psi°$ of { Specimen, (*No. 8*) deg. / Circuit, (*No. 9*) deg. }
Power factor of Specimen, (*No. 10*) %.
Dielectric Constant, K = (*No. 13*).
Phase difference × Dielectric Constant, $\Psi° \times K$ = (*No. 14*).

Galvanometer.				Resistance Inserted, R_1	$2\sqrt{\frac{d_o}{d_1}}-1$	Total Equiv. Resistance, R	Equiv. Res. of C_x, R_x
Zero Deflection.	Maximum Deflection.	True Deflection.	Mean Deflection.				
0.30	41.70	41.40	41.40	*A* 0	.....	.....	22.08
0.32	15.00	14.68	14.69	*B* 18.01	0.679	26.53	.....
0.32	41.70	41.38	41.40	*A* 0	.....	.....	.....
0.33	13.67	13.34	13.36	*C* 20.21	0.761	26.56	.....
0.33	41.70	41.37	41.37	*A* 0	.....	.....	.....
0.38	12.81	12.43	12.43	*D* 21.84	0.825	26.48	.....
0.38	41.79	41.41	.....	*A* 0	.....	.....	.....
0.36	13.75	13.39	.....	*C* 20.21	.....	Ave.	.....
0.37	41.77	41.40	.....	*A* 0	.....	.....	.....
0.39	15.09	14.70	.....	*B* 18.01	.....	26.52	.....
0.49	41.71	41.22	41.23	*A* 0	.....	.....	.....
0.52	17.35	16.83	16.84	*B* 2.51	0.565	4.44	.....
0.50	41.74	41.24	41.24	*A* 0	.....	....	.....
0.52	14.91	14.39	14.40	*C* 3.08	0.692	4.45	.....
0.50	41.77	41.27	41.27	*A* 0	.....	....	.....
0.52	11.42	10.90	10.90	*D* 4.20	0.947	4.43	.....
0.53	41.78	41.25	.....	*A* 0	.....	.....	.....
0.53	14.93	14.40	.....	*C* 3.08	.....	Ave.	.....
0.53	41.77	41.24	.....	*A* 0	.....	....	.....
0.55	17.39	16.84	.....	*B* 2.51	.....	4.44	.....

entry in Column No. 2 and the ohmic resistance of the resistor as a second entry in Column No. 5. Assuming the copper link to be A and three non-inductive resistors of increasing resistance values to be B, C, and D, insert the several links (A, B, C, and D) on the grounded side of the switch between wells 2 and 4 in the order AB, AC, AD, AC, AB. Record each zero error and maxi-

mum deflection in the proper place in Columns Nos. 1 and 2 and the value of resistance inserted in the circuit in Column No. 5. Remove the links from the wells 1 to 3 and 2 to 4 and insert copper links between 3 and 5 and between 4 and 6. Move the generating circuit about 50 cm. away from the standard inductor L, being careful not to change the setting of any of the variables in the generating circuit. Check the wave frequency of the generating circuit. Tune the measuring circuit (composed of the standard condenser C_s, standard inductor L, the thermo-element T and the galvanometer G) to the generating circuit by varying the setting of the standard condenser C_s. Change the coupling between L_g and L to give about 40 cm. galvanometer deflection on the 50-cm. scale. Tune the measuring circuit very carefully but do not vary any controls on the generating circuit. Read the setting of the standard condenser C_s and record in Space No. 3. If more than one standard condenser is available in the laboratory, record the number of the

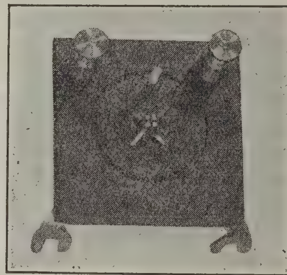

FIG. 11.—Thermo-element.

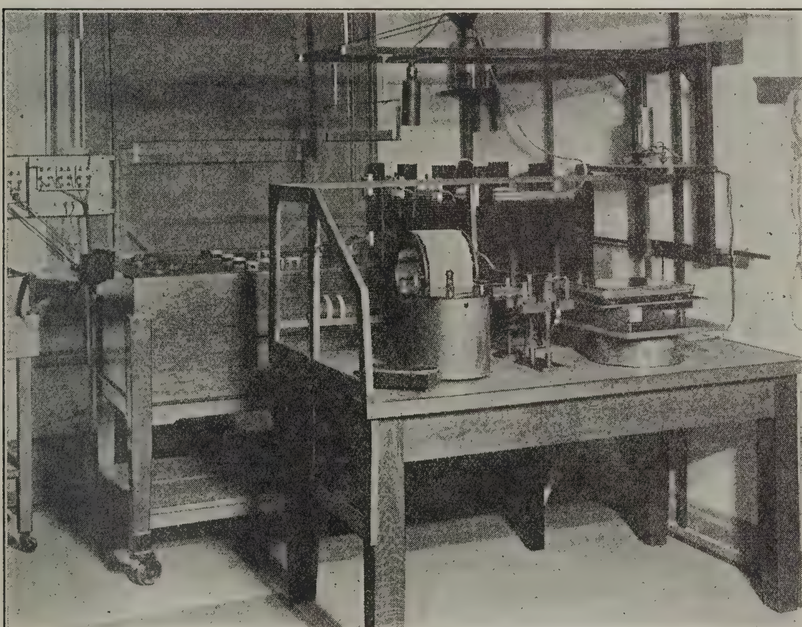

FIG. 12.—Complete Apparatus as Installed in Radio Laboratory, U. S. Bureau of Standards.

condenser used in Space No. 4 for the purpose of identification. Remove the copper link from between wells 4 to 6, read the galvanometer zero deflection and record the value in the first column of the data sheet (see 0.049, Column

No. 1). Reinsert the copper link and read the maximum galvanometer deflection, record the numerical value in Column No. 2. Proceed in this manner, as above described, inserting resistors in the order *AB*, *AC*, *AD*, *AC*, *AB*.

The data in the body of the data sheet may now be computed. Column No. 3 is obtained by subtracting the zero deflection from the corresponding maximum deflection. Thus 41.70 − 0.30 = 41.40, the true deflection, or 15.00 − 0.32 = 14.68. Column No. 4 is obtained by averaging; that is, the two *A* deflections which precede the two *B* deflections, the two *B* deflections, the two *A* deflections which precede the two *C* deflections and the two *C* deflections are averaged. Thus, the average of 41.40 and 41.40 is 41.40 and of 14.68 and 14.70 is 14.69. As has been stated, phase difference

$$\psi^\circ = 0.1079 \frac{RC}{\lambda}$$

The equivalent resistance *R* is computed in three steps and recorded in Column No. 8. By one measurement the equivalent resistance (R_x) of the condenser

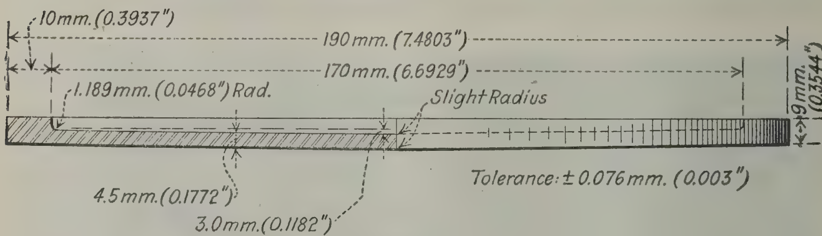

FIG. 13.—Molded Test Specimen.

C_x plus the resistance (R_c) of the remainder of the circuit, including the inductor *L*, is determined. By another, the equivalent resistance (R_s) of the standard condenser C_s and the resistance (R_c) of the remainder of the circuit, including the inductor *L*. Since in each case the remainder of the circuit is the same and the standard condenser C_s is considered to have negligible resistance, the difference of the resistance values will be the equivalent resistance of the specimen condenser C_x. That is, the resistance of the condenser C_x plus the resistance of the circuit is $R_x + R_c$, while the resistance of the standard condenser C_s plus the resistance of the circuit is $R_s + R_c$. R_s is negligible, so $R_s + R_c$ is equivalent to R_c. $(R_x + R_c) - R_c = R_x$ = the equivalent resistance of C_x.

The combined equivalent resistance of the specimen condenser and its attached circuit is computed first.

$$\text{The equivalent resistance } R = \frac{R_1}{\sqrt[2]{\dfrac{d_0}{d_1}} - 1}$$

where R_1 = resistance inserted,
d_1 = galvanometer deflection with R_1 inserted,
d_0 = galvanometer deflection with no resistance inserted.

The denominator is solved first and its values inserted in Column No. 6, while the values of R are inserted in Column No. 7. Thus, the values in Column No. 6 are

$$\sqrt[2]{\frac{41.40}{14.69}} - 1 = 0.679, \sqrt[2]{\frac{41.40}{13.36}} - 1 = 0.761, \text{ and } \sqrt[2]{\frac{41.37}{12.43}} - 1 = 0.825.$$

The equivalent resistance R (Column No. 7) is obtained by dividing the ohmic resistance of the resistors B, C, and D by their respective denominators as shown in the above equation. Thus, the values of R are

$$\frac{18.01}{0.679} = 26.53, \quad \frac{20.21}{0.761} = 26.56, \quad \frac{21.84}{0.825} = 26.48.$$

The same procedure is followed to determine the resistance of $R_s + R_c$. As shown in the data sheet, these values are found to be 4.44, 4.45 and 4.43 ohms. The next step is to average each group of three resistances. The equivalent resistance of the specimen condenser C_x is the difference between these averages, or 26.52 – 4.44 = 22.08 ohms. The next step is to fill in Spaces Nos. 5, 6 and 7 by referring to the calibration charts of the wave meter and the standard condenser. With these values available, the phase difference, or power factor of the specimen, may be computed. As has been previously stated, phase difference expressed in degrees is

$$\psi^\circ = 3.60\, RCf \times 10^{-7}$$

or

$$= 0.1079 \frac{RC}{\lambda}$$

Assuming the specimen capacity (C) to be 235 micromicrofarads, the wave frequency (f) to be 1000 *kc* per sec. (wave length 300 meters), then the phase difference of the specimen is

$$\psi^\circ = 3.60 \times 22.08 \times 235 \times 1000 \times 10^{-7} = 1.87 \text{ deg.}$$

or

$$= \frac{0.1079 \times 22.08 \times 235}{300} = 1.87 \text{ deg.}$$

This value is inserted in Space No. 8 while the phase difference of the circuit is computed and inserted in Space No. 9. Thus the phase difference of the circuit equals:

$$3.60 \times 4.44 \times 235 \times 1000 \times 10^{-7} = 0.38 \text{ deg.}$$

or

$$\frac{0.1079 \times 4.44 \times 235}{300} = 0.38 \text{ deg.}$$

For the small values of phase difference usually found in the better types of electrical insulating material,

$$\text{power factor in per cent} = 1.75 \times \psi^\circ.$$

This quantity is computed and entered in Space No. 10. The thickness and area of the specimen may now be measured and the values written in Spaces Nos.

11 and 12, respectively. The thickness (T) should be the average of at least six micrometer readings. Having determined the thickness (T) and the area (S), and the capacity (C) (Spaces Nos. 7, 11 and 12), the dielectric constant may be computed from the formula previously given. Assuming T and S to be 0.45 cm. and 284 cm². respectively,

$$K = \frac{CT}{0.0885\ S} = \frac{235 \times 0.45}{0.0885 \times 284} = 4.2.$$

The computed value of K is written in Space No. 13. For the better comparison

TABLE II.—SAMPLE REPORT.

Date of Test, *May 10, 1922.* Specimen Number, *1.*
Specimen submitted by *A B C Co.* Number submitted, 1. Date, *May 1, 1922.*
Base or Filler, *Wood flour.* Binder, *X Y Z Resin.* Grade, *No. 2.* Color, *Natural.*
Average thickness, *0.443* cm.,. . . . . .in. Room temperature, *21*° C.,. . . . .°F.
Relative humidity, *54%*.

Wave Frequency, f, kilocycles per sec.	Wave Length, λ, meters.	Computed Values.				Remarks.
		Phase Difference, $\psi°$, deg.	Power Factor, per cent.	Dielectric Constant, K	Product of $\psi° \times K$	
1071.0	280	2.04	3.57	5.20	10.6	As received.
492.0	610	1.92	3.36	5.28	10.1	
197.4	1520	1.91	3.34	5.34	10.2	
113.6	2640	1.86	3.25	5.37	10.0	
95.0	3160	1.84	3.22	5.40	9.9	
1027.0	292	2.12	3.71	5.40	11.4	After water treatment.
482.5	622	2.02	3.54	5.48	11.1	
195.5	1534	2.01	3.52	5.54	11.1	
112.5	2666	1.96	3.43	5.57	10.9	
93.8	3200	1.94	3.39	5.60	10.9	

of electrical insulating materials for radio use, both the phase difference and the dielectric constant should be considered. Space No. 14 has been left for the numerical product of phase difference expressed in degrees and the dielectric constant. Thus,

$$\text{Product} = \psi° \times K = 1.87 \times 4.2 = 7.86.$$

Report.—A convenient torm for the report is shown in Table II and a summary for 300 and 3000 meters is shown in Table III.

TABLE III.—SAMPLE SUMMARY.

Values of phase difference and dielectric constant, at 300 and 3000 meters wave length as read from curves, Fig. 14.

Wave Frequency.	Phase Difference, $\psi°$, deg.	Dielectric Constant, K	Product of $\psi° \times K$
AS RECEIVED, AT 21° C.			
100 *kc* per sec. 3000 meters	1.84	5.39	9.92
1000 *kc* per sec. 300 meters	2.02	5.21	10.53
Average	1.93	5.30	10.23
AFTER 48 HOURS IN WATER AT 21° C.			
100 *kc* per sec. 3000 meters	1.94	5.60	10.86
1000 *kc* per sec. 300 meters	2.11	5.40	11.40
Average	2.02	5.50	11.13

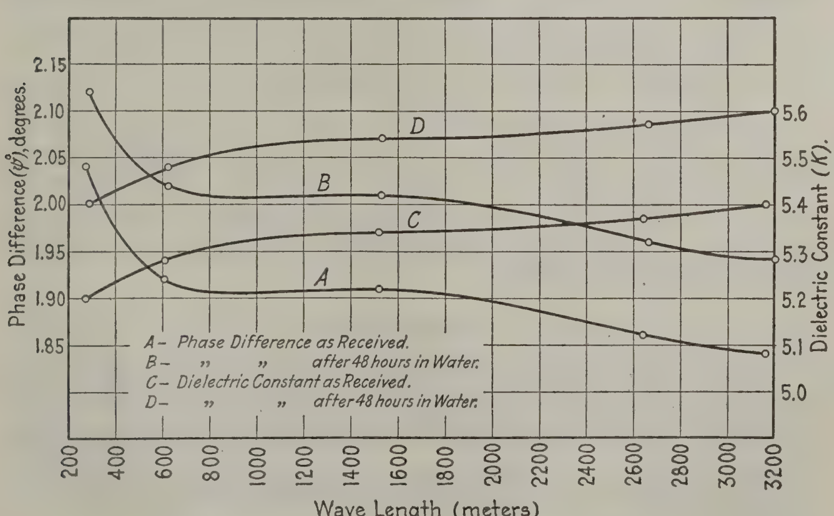

FIG. 14.—Curves for Test Specimen No. 1.

TENTATIVE METHOD OF TESTING ELECTRICAL INSULATING MATERIALS FOR VOLTAGE EFFECTS AT RADIO FREQUENCIES[1]

Serial Designation: D 175 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

Issued, 1923; Revised, 1925.

Scope.

1. This method of test is intended to determine the dielectric failure of electrical insulating materials at radio frequencies.[2]

The failure under radio frequency stress may take the form of charring, buckling, cracking, blistering, softening or chemical decomposition. Failure through the material is not abrupt and it, therefore, requires a certain amount of judgment on the part of the operator to decide just what constitutes failure in any particular case.

When a sheet of insulating material is tested between two metal electrodes passing through it, the heat generated increases with the thickness of the material, while the radiating surface remains nearly constant. Consequently, as the thickness of the sample is increased, the voltage at which failure occurs will decrease. As sufficient data are not available to allow results to be translated from one thickness to another, it is necessary that all comparative tests be made on samples of the same thickness. Hence, in the standard method given below, a definite thickness of sample is specified. This method, of course, is applicable to other thicknesses of material, but when so used the results should be compared only with similar tests made on like thicknesses of material.

I. APPARATUS

Generating Circuit.

2. Any type of generator having an output of 500 watts or more and generating continuous waves of 100 and 1000 kilocycles per second, respectively, and at voltages up to 5000 or 10,000 volts, depending upon

[1] Criticisms of this Tentative Method are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

[2] A high frequency dielectric endurance test is being developed and will later be included in this tentative standard.

the class of materials tested, is satisfactory. If desired, a circuit similar to that shown in Fig. 1 may be used.

3. The voltage may be measured by means of the current through a shielded radio frequency ammeter in series with a small shielded air **Voltmeter.**

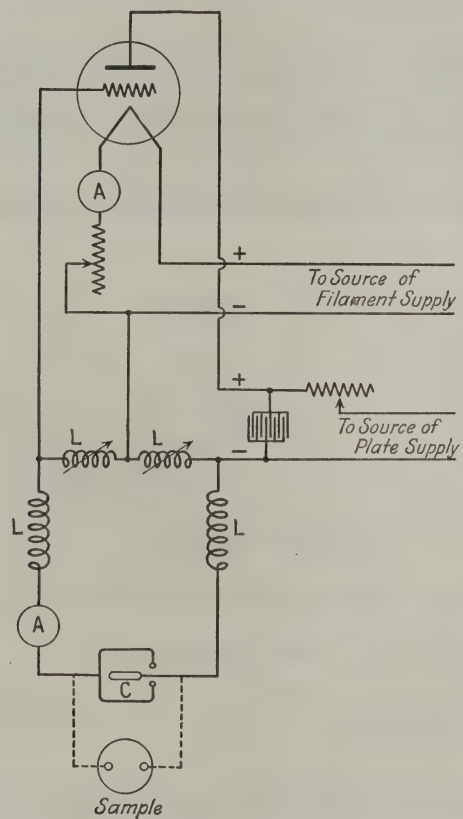

FIG. 1.—Schematic Diagram of Circuit for Determination of Voltage Effects.

condenser of known capacity across the test terminals or by means of an electrostatic voltmeter designed to withstand radio frequency potentials. Suitable scales shall be provided so that reasonable accuracy will be obtained when measuring any testing voltages up to the maximum desired.

If the ammeter method is used:

$$E \text{ (in volts)} = \frac{16I \times 10^7}{fC}$$

where I = current in amperes;
f = frequency in kilocycles per second;
C = capacity of air condenser in micro-microfarads.

NOTE.—In the above formula, the value 16 is an approximation, 15.92 being the exact value.

Frequency Meter. 4. The frequency meter or wave meter may be of any commercial design covering the required frequency or wave lengths and accurate to within ± 10 per cent over the wave frequency range used.

Electrodes. 5. (*a*) The electrodes shall be of brass and shall be clean and polished.

(*b*) The electrodes shall have the dimensions shown in Fig. 2.

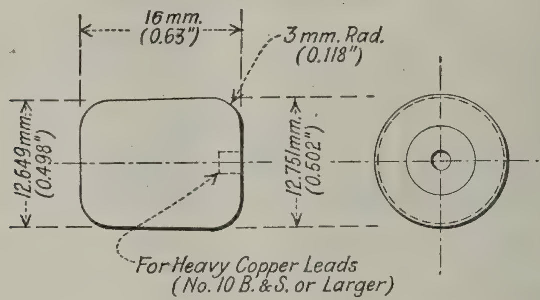

FIG. 2.—Showing Dimensions of Electrodes.

II. TEST SPECIMENS

Test Specimens. 6. (*a*) The specimens shall be representative of the material to be tested, care being taken to select material free from abnormal defects.

(*b*) The specimens shall be 6.35 mm. (0.25 in.) in thickness and may be of any convenient size or shape provided that the center electrode (see Fig. 3) is at least 57.5 mm. (2.264 in.) from the outside edge at all points. Other thicknesses may be used but the results therefrom should be used strictly for comparative purposes as explained under Section 1. If any samples are milled to reduce the thickness, care should be taken not to tear, chip or otherwise change the character of the material. Four holes 12.7 mm. (0.5 in.) in diameter, shall be provided in each specimen, one in the center and three equally spaced in a circle of 25.4 mm. (1.000 in.) radius as shown in Fig. 3.

III. PROCEDURE

7. (*a*) Two specimens shall be tested in the condition received at normal room temperature of about 20° C. (68° F.), one of these at 100 kilocycles per second (3000 meters wave length) and the other at 1000 kilocycles per second (300 meters wave length). **Surrounding Medium and Wave Frequency.**

(*b*) Similarly, an additional two specimens shall be tested respectively, at the frequencies specified in Paragraph (*a*) after the specimens have been immersed in distilled water at a temperature of about 20° C. (68° F.) for 48 hours. After removing the specimens from the water, the surface water shall be wiped off with a clean absorbent cloth and the specimens then allowed to stand in the room away from drafts and

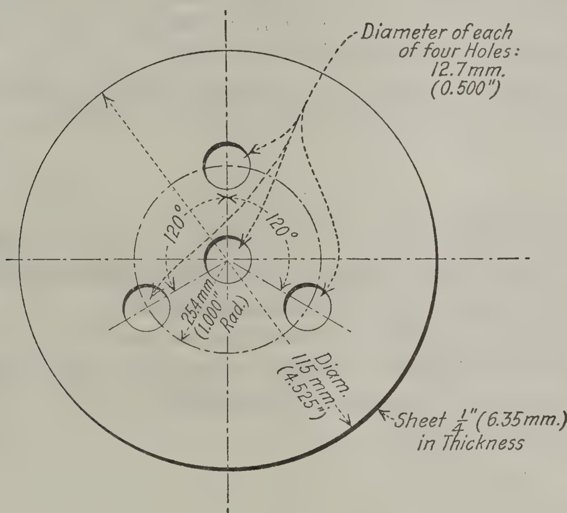

FIG. 3.—Test Specimen.

direct sunlight for 5 minutes. The holes shall be drilled before immersion.

8. One electrode (Fig. 2) shall be inserted in the center hole in a specimen and a similar electrode in one of the other three holes. The electrodes must be a tight fit, otherwise corona may form at comparatively low voltage and destroy the material. **Position of Electrodes in Specimens.**

9. The electrodes shall then be connected to the test terminals of the radio-frequency generator. One side of the generator shall be grounded, and this terminal shall be connected to the center electrode of the specimen. The reason for this is that the failure generally occurs around the high potential terminal and this will allow the two subsequent tests to be made between the center and the other two holes, the center not being destroyed. **Connection of Electrodes to Test Terminals.**

Application of Voltage. 10. For materials which ordinarily fail below 3000 volts, the voltage shall be raised in steps of 500 volts. For materials which ordinarily fail above 3000 volts, the voltage shall be raised in steps of 1000 volts. Wherever possible, the initial voltage chosen shall be such that at least three voltage steps will be required to cause failure of the material.

Test Period. 11. The voltage at each step shall be impressed for two minutes, the testing period being counted from the instant when the prescribed voltage is reached, and the time required to reach this voltage not exceeding $\frac{1}{4}$ minute. Whenever samples thicker than that specified are used, this testing period should be changed to three minutes.

Number of Tests. 12. Tests shall be made between the center hole and each of the other three holes of each specimen, the specimens being allowed to cool to room temperature after each test.

The average of these three voltages shall be considered as the dielectric strength of the specimen under the conditions of the tests.

IV. REPORT

Report. 13. The report of the tests shall include:

(*a*) The description of the material, *i. e.*, base or filler, binder, grade, color and nature of surface of specimen between electrodes;

(*b*) Room temperature and relative humidity;

(*c*) Voltage at which each dielectric failure occurs, average voltage, duration of application of final voltage, and wave frequency.

(*d*) Thickness of specimen at electrodes;

(*e*) Nature of failure.

APPENDIX

SAMPLE RECORD OF TEST

Date of Test,............ Test No.,............
Specimens submitted by,............ Number Submitted,............ Date,............
Base or Filler,............ Binder,............ Grade,............ Color, ...
Dry Bulb,............° C. Wet Bulb,............° C. Relative Humidity,............%

DIELECTRIC FAILURE TEST

	Specimen Number			
	1	2	3	4
Thickness of specimen at electrodes (mm.)...	......	......	......	......
Distance between electrode edges (mm.)......	......	......	......	......
Duration of immersion tests (hours)..........	......	......	......	......
Temperature of water (deg. Cent.)............	......	......	......	......
Frequency (kilocycles per second)............	......	......	......	......
Wave length (meters)......................	......	......	......	......
Initial voltage.............................	......	......	......	......
Voltage at failure:				
First test	......	......	......	......
Second test	......	......	......	......
Third test	......	......	......	......
Duration of Application of Final Voltage (seconds):				
First test.............................	......	......	......	......
Second test............................	......	......	......	......
Third test.............................	......	......	......	......
Condition of material after failure:				
First test	......	......	......	......
Second test	......	......	......	......
Third test	......	......	......	......

TENTATIVE METHODS OF TESTING CABLE SPLICING AND POTHEAD COMPOUNDS[1]

Serial Designation: D 176 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1924, 1925.

Material Covered. 1. These tests are intended to cover compounds used in cable splices and potheads.

I. PHYSICAL TESTS

Physical Tests. 2. Each of the following tests shall be made in accordance with the method of test applicable, of the American Society for Testing Materials, as indicated:

(*a*) *Melting Point:* Tentative Method of Test for Melting Point of Petrolatum (Serial Designation: D 127 – 24 T).[2]

(*b*) *Softening Point*, for compounds having no definite melting point: Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (Serial Designation: D 36),[3] or

Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (Serial Designation: D 61).[3]

The method used should be indicated in the report.

(*c*) *Flash and Fire Points:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (Serial Designation: D 92).[3]

(*d*) *Evaporation:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (Serial Designation: D 6).[3]

(*e*) *Viscosity*, Temperature range from melting point to 150° C.: Saybolt Furol, Tentative Methods of Test for Viscosity of Petroleum Products and Lubricants (Serial Designation: D 88 – 25 T).[4]

(*f*) *Penetration Test*, Temperature range from melting or softening point to −25° C.: Standard Method of Test for Penetration of Bituminous Materials (Serial Designation: D 5).[5]

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

[2] See p. 341.

[3] 1924 Book of A.S.T.M. Standards.

[4] See p. 358.

[5] A.S.T.M. Standards Adopted in 1925.

3. To determine the brittleness of the material, the compound shall be spread on a piece of sheet metal in a layer $\frac{1}{16}$ in. in thickness. It shall then be submerged in water having a temperature at least 20° F. above the expected breaking point. It shall then be cooled 5° F. and held at this temperature for 5 minutes. The cooling shall then be continued in increments of 5° F. At each temperature interval the compounds shall be tested with the blade of a knife, inserting the point with the blade as nearly parallel with the sheet metal as is practicable, and pulling the compound from the sheet metal as rapidly as possible by rotating the blade about its back as an axis. The temperature at which the compound no longer stretches but snaps, shall be considered the breaking point. **Brittleness.**

II. DIELECTRIC STRENGTH TEST

4. (*a*) This test is intended to determine the dielectric strength of cable splicing and pothead compounds under voltage stresses of short duration. It is of value as a comparative test but does not indicate the permanent dielectric strength of these compounds. **Dielectric Strength.**

(*b*) The dielectric strength of this class of material varies with the temperature. It is, therefore, recommended that a sufficient number of tests be made at different temperatures to establish the momentary dielectric strength - temperature curve from −25° C. to +100° C.

5. A representative sample shall be taken from the original package, melted and poured directly into the testing container. Care should be used in melting and pouring the compound not to overheat it nor to entrap air. The electrodes should be heated to the approximate compound temperature before filling the container. **Sample.**

6. (*a*) In the case of materials which are hard at room temperature, they shall be tested between hemispherical electrodes embedded therein which are $\frac{1}{2}$ in. in diameter separated by a gap of 0.1 in. **Electrodes.**

NOTE.—A convenient form of apparatus for holding the electrodes and compound is described in the Appendix.

(*b*) In the case of materials which are soft at room temperature, they shall be tested between polished brass or copper circular disk electrodes, 1 in. in diameter and having a square edge, the electrodes to be placed with their axes horizontal and coincident and with a gap of 0.1 in. between their adjacent faces.

NOTE.—This test can be made with the apparatus normally used for testing transformer and switch oils. (See the Standard Methods of Testing Transformer and Switch Oils (Serial Designation: D 117) of the American Society for Testing Materials.[1])

[1] 1924 Book of A.S.T.M. Standards.

Testing Transformer. Voltage Control and Measurement. 7. The testing transformer used, methods of voltage control and measurement shall be as outlined in Section 2 of the Tentative Methods of Testing Sheet and Tape Insulating Materials for Dielectric Strength (Serial Designation: D 149 – 24 T) of the American Society for Testing Materials.[1]

Number of Specimens. 8. Five specimens at a given temperature shall be tested and the average value taken as the momentary dielectric strength of the compound at that temperature.

Report. 9. The report shall include the following:

(*a*) The momentary dielectric strength – temperature curve;

(*b*) The individual values of the puncturing voltage at the various temperatures.

III. COEFFICIENT OF EXPANSION

Apparatus. 10. Calibrated flasks of Pyrex or quartz glass holding approximately 250 cc. to the zero mark, having necks 1 cm. in internal diameter and graduated for 25 cc. in 0.1-cc. divisions, total capacity approximately 275 cc., shall be used.

NOTE.—If it is necessary to use a small sample, then a Cassia flask holding 100 cc., with neck graduated for 10 cc. in 0.1-cc. divisions, may be used.

11. The cylindrical oil bath for heating the sample shall be approximately 10 in. (25.4 cm.) in inside diameter and 20 in. (50.8 cm.) in inside depth with a false bottom 1 in. from the bottom and shall have provision for circulating the oil.

NOTE.—This bath may be made as follows: A shell about 10 in. (25.4 cm.) in inside diameter and 20 in. (50.8 cm.) in inside depth is electrically heated by winding the shell with resistance wire and covering with five layers of asbestos paper. A motor-droven paddle at the bottom will give effective oil circulation.

12. A lead collar to be hung on the flask during test to prevent the oil currents of the bath from moving the flask.

Calibration. 13. The capacity of the flask at the zero point and several points on the scale shall be determined by filling the flask with distilled water at a known temperature and weighing.

Procedure. 14. The flask shall first be warmed slightly, then partly filled with melted compound and placed in the bath which shall then be brought to the highest temperature of the range over which the test is to extend. The flask shall then be filled to within the last cc. marked on the neck. The bath shall be held at this temperature for not less than 30 minutes (principally to remove air bubbles) and the whole shall then be slowly cooled to room temperature (10 to 12 hours.) Before starting the test, the flasks shall be examined for the

[1] See p. 650.

the presence of cavities or irregular contraction of the compound. Some compounds, after cooling below the liquid state, tend to stick to the sides of the neck of the flask. In that case it is necessary to gradually warm the neck and flow the compound to meet the rest, after which the flask is placed in the bath for an hour or so to insure equalizing the temperature.

With the compound satisfactorily placed in the flasks at the lowest temperature, the height of the column in the neck shall be read and the bath then slowly heated. Readings shall be taken at 5° C. intervals, holding the bath as constant as possible at each point until no more expansion occurs at that point, the procedure being repeated for each point until maximum temperature is reached.

Precautions.

15. During the test, temperature readings shall be taken at top and bottom of the bath to detect any variation.

Until the compound is completely liquefied, intervals between readings should be three to four hours, especially between 35 and 55° C.

After becoming liquefied, the time necessary for obtaining a reading may be reduced to about 30 minutes.

Report.

16. (*a*) After securing the readings over the temperature range desired, a curve shall be plotted from the temperature and flask readings from which the coefficient of expansion shall be calculated from the formula:

$$\frac{V_1 - V}{(T_1 - T)\ V} + C$$

where V = original volume;
V_1 = volume at higher temperature;
T = original temperature;
T_1 = higher temperature;
C = constant = coefficient of expansion of glass container.

(*b*) The coefficient shall be calculated for three ranges as follows:

(1) From the minimum temperature at which the measurement was made to 10° C. below the melting point. This is intended to give the average coefficient for the solid condition.

(2) From 5° C. above the melting point to 100° C. This is intended to give the average coefficient for the liquid condition.

(3) From the minimum temperature at which a measurement was made to 100° C.

In reporting the coefficient of expansion the corresponding temperature range shall be indicated in each case.

APPENDIX

Because of the great difficulty in removing most cable splicing and pot head compounds from the container and from the electrodes, an arrangement was devised sufficiently inexpensive to permit of its being discarded wholly or in part after the test. That is, each specimen with its container can be entirely discarded without undue expense.

One form of container consists of a small rectangular box about 2½ in. (6.4 cm.) in length and 1½ in. (3.8 cm.) square, the ends of which are dry wood about ¼ in. in thickness. The sides and bottom are formed from a piece of thin, hard fiber tacked to the wooden end pieces. The fiber is partially cut at the corners which permits making a sharp, square bend.

Each electrode is mounted on the end of a piece of straight copper wire, No. 10 A.W.G., which passes through a hole at the center of the wooden end

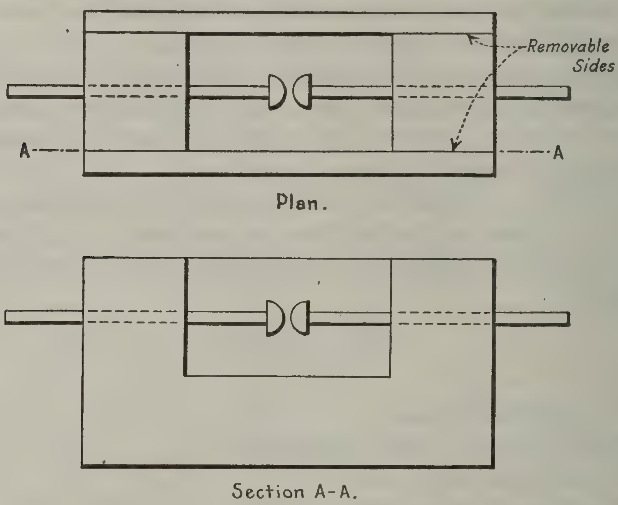

FIG. 1.—Container, Showing Electrodes in Place for Dielectric Test of Cable Splicing and Pothead Compounds.

pieces. These holes are drilled in one operation so that they are in the same straight line. They are made slightly smaller so that the rods fit tightly and are therefore not readily moved.

Fig. 1 shows a sketch of a container in which the ends and the bottom are formed from one block of thoroughly dried wood which has been boiled in paraffin, the sides being pieces of thin, hard fiber tacked to the wood.

In the construction of these containers, it is obviously important that the electrodes be reasonably co-axial and that the gap remain constant.

In making tests at high voltage it will probably be found necessary to immerse the container with its specimen in oil in order to prevent flash-over between the exposed ends of the electrode rods. The ends of the rods can be bent to form hooks, by means of which the specimen holder can be readily suspended in a jar of oil.

TENTATIVE METHODS OF TESTING UNTREATED INSULATING PAPER[1]

Serial Designation: D 202 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

1. These methods apply to untreated paper which is to be used as an electrical insulator or as a constituent of a composite material used for electrical insulating purposes. **Material Covered.**

I. SAMPLING

2. (*a*) From shipments consisting of rolls, a sample of at least 1 sq. yd. in area, cut across the entire width of the roll, shall be taken from every tenth roll in the shipment. From other shipments such as sheets or pads of tape, samples of equivalent area shall be taken in such a manner as to be equally representative of the shipment. In no case shall less than three samples be taken. **Sampling.**

NOTE.—It is recommended that several turns of paper from each roll sampled be torn off and discarded before the samples are taken.

(*b*) The tests for physical properties shall be made upon each sample.

(*c*) The tests for acidity,[2] moisture content, size,[2] ash and for identification of fibers[2] shall be made in duplicate upon portions taken from a composite sample. This composite sample shall be made up of a sufficient number of small pieces, each of approximately 6.5 sq.cm. (1 sq. in.) in area taken in nearly equal amounts from each of the samples taken in accordance with Paragraph (*a*).

(*d*) The required number of samples shall be taken at equal intervals across the sheet, the two end samples being taken at the edges of the sheet after removing any damaged paper. When it is

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

[2] The tests for acidity, size, identification of fibers, and folding endurance do not appear in these methods but will be included as soon as the tests are developed by the committee.

not possible to secure the required number of specimens in this manner in going once across the sheet, the selection across the sheet shall be repeated as many times as necessary until the required number of specimens is secured.

II. CONDITIONING SAMPLES

Conditioning.

3. Samples shall be conditioned in air maintained at a relative humidity between 60 and 65 per cent as measured with a sling psychrometer or its equivalent. The temperature of the air shall be maintained as constant as possible at some temperature between the limits of 20 and 30° C. (68 and 86° F.). The samples should remain in the conditioned air for not less than 4 hours prior to the tests and should be supported so as to allow a free circulation around each sample. The following physical tests shall be made in the conditioned air: tensile breaking strength, tearing strength, bursting strength, folding endurance,[1] air permeability,[1] and thickness.

III. MOISTURE CONTENT

Apparatus.

4. A weighing bottle, evaporating dish, thermometer, constant-temperature oven, chemical balance and desiccator are necessary for the test. The weighing bottle should be of convenient size, about 65 mm. (2.56 in.) in height and 45 mm. (1.77 in.) in diameter with a wide mouth provided with a ground-glass stopper. The chemical balance should be sensitive to 0.1 mg.

Speci en.

5. The moisture content figure is used to calculate the percentage of acidity, size, and ash of the paper to the bone-dry basis. It shall be determined on 2 to 5-g. samples selected in accordance with Section 2 (*c*).

If the moisture content of the paper as received at the purchaser's works is desired or at any other stated time, it shall be made on the sample taken at the definitely stated time by cutting small pieces of approximately 6.5 sq. cm. (1 sq. in.) in area from the required number of rolls or pads of tape in accordance with Section 2 (*a*) and immediately placed into an air-tight container.

NOTE.—Since paper adjusts itself, as regards moisture content, to the humidity of the surrounding atmosphere in a very short time (2 or 3 minutes), special care must be exercised to transfer rapidly the sample from the roll or bundle to the container and from the container to the weighing bottle.

Procedure.

6. The sample before drying shall be weighed in the tared bottle with the stopper in place. The bottle shall then be placed in the oven at 100 to 105° C. (212 to 221° F.), the stopper removed and laid alongside of the bottle and the contents transferred to the drying

[1] The tests for acidity, size, identification of fibers, and folding endurance do not appear in these methods but will be included as soon as the tests are developed by the committee.

dish. After one hour, while still in the oven, the sample shall be replaced in the weighing bottle, and the bottle stoppered and transferred to the desiccator. The stopper should be removed while the bottle cools. When the specimen and the bottle have cooled to room temperature, the stopper shall be replaced and the bottle with its contents weighed. This process shall be repeated at intervals of one hour until the difference in weight between two successive weighings is not more than 0.2 per cent of the weight of the specimen.

NOTE.—The weighing bottle or sample should not be touched with the fingers during this test.

Report.

7. The moisture content shall be expressed:
(*a*) as a percentage of the weight of the dry sample, and
(*b*) as a percentage of the weight of the undried sample.

IV. THICKNESS

Apparatus.

8. A micrometer of the spring actuated, dial type shall be used. The plunger shall be capable of being raised by the application of an upward pressure to it. The plunger surface shall be circular in shape and 12.7 mm. (0.50 in.) in diameter.

In addition to these requirements, the instrument shall conform to the following:

(*a*) The pressure applied to the plunger necessary to move the pointer from zero to a positive reading shall be not greater than 1135 g. (40 oz.).

(*b*) The pressure applied to the plunger necessary to just prevent movement of the pointer from zero to a lower reading shall be not less than 455 g. (16 oz.).

(*c*) If the instrument is provided with a device for controlling the speed of descent of the plunger, the latter shall not strike the anvil with an impact pressure greater than 1135 g. (40 oz.).

(*d*) The diameter of the dial shall be approximately 15 cm. (6 in.) and shall be graduated preferably in divisions indicating a thickness of 0.0127 mm. (0.0005 in.) and in no case greater than 0.0254 mm. (0.001 in.). Graduations indicating a thickness of 0.0254 mm. (0.001 in.) shall be at least 3 mm. (0.12 in.) apart.

(*e*) Measurements made on standard steel thickness gages shall be within the following tolerances:

Intervals	Permissible Deviation of Reading from Actual Thickness of Standard Steel Gage
0 to 0.25 mm. (0 to 0.01 in.) incl.	±0.0025 mm. (0.0001 in.)
Over 0.25 mm. to 1.02 mm. (0.01 in. to 0.04 in.) incl.	±0.0051 mm. (0.0002 in.)
Over 1.02 mm. to 3.05 mm. (0.04 in. to 0.12 in.) incl.	±0.0102 mm. (0.0004 in.)

(*f*) Convenient means shall be provided for setting the pointer to the zero position.

(*g*) The micrometer shall be tested periodically as outlined above and if it does not conform to these requirements its mechanism should be readjusted.

Specimens. 9. Specimens shall consist of the original samples obtained in accordance with Section 2 (*a*).

Methods. 10. At least five readings shall be taken at regular intervals across the entire width of each specimen. When measuring paper less than 0.05 mm. (0.002 in.) in thickness, a sufficient number of layers to give a reading on the scale of not less than 0.13 mm. (0.005 in.) shall be used.

Report. 11. The average, maximum and minimum thicknesses obtained on each specimen shall be reported.

V. TENSILE BREAKING STRENGTH

Apparatus. 12. (*a*) A testing machine suitably designed for testing paper shall be used. The machine shall preferably be power driven.

(*b*) The capacity of the machine shall not exceed 113 kg. (250 lb.).

(*c*) The machine shall be graduated to read 1 lb. or 1 kg. or less per scale division for testing paper breaking at 22.7 kg. (50 lb.) or over, and to 0.5 lb. or 0.5 kg. or less for testing paper breaking under 22.7 kg. (50 lb.).

Specimens. 13. From each of the samples selected in accordance with Section 2 (*a*), specimens shall be cut at least ten in the machine direction and if practicable, ten in the cross-machine direction. The specimens shall not exceed 2.54 cm. (1 in.) in width and shall be 25.4 cm. (10 in.) in length with clean-cut edges.

Procedure. 14. (*a*) The ratio of the clearance distance between jaws to the width of the specimen shall be not less than 5:1 nor more than 10:1.

(*b*) The rate of travel of the movable jaw shall be constant. It shall preferably be 30.5 cm. (12 in.) per minute, but it may be within the limits of 28 cm. (11 in.) and 33 cm. (13 in.) per minute provided it is constant.

(*c*) All readings obtained when the paper breaks at or in the jaws shall be rejected.

Report. 15. (*a*) The results of the machine-direction specimens and the cross-machine-direction specimens shall be reported separately.

(*b*) The results shall be reported in kilograms or pounds together with the width of the specimen in centimeters or inches and also the average thickness.

(*c*) The maximum, minimum, and average breaking load shall be reported for the machine-direction and the cross-machine direction.

VI. TEARING STRENGTH

16. The testing machine shall be of the pendulum impulse type so designed as to produce a tear approximately 4.2 cm. (1.66 in.) long. The knife mounted on the machine to cut the slit for the tear shall be maintained sharp. **Apparatus.**

17. The specimens shall be taken from the original samples obtained in accordance with Section 2 (*b*) and shall be cut 6.3 cm. (2.5 in.) in width and at least 6.3 cm. (2.5 in.) in length. Enough specimens shall be cut so that at least five readings in the machine direction and five readings in the cross-direction, if possible, can be obtained for each original sample. **Specimen.**

18. Enough paper shall be torn so that the readings on the machine shall be not less than 10 nor more than 20 g., unless one paper alone yields a reading greater than 20 g., in which case only one paper shall be torn at one time. Readings obtained when a tear deviates more than 6.3 mm. (0.25 in.) from a straight line, shall be rejected. The readings obtained shall be multiplied by the instrument constant corresponding to the number of sheets torn. **Method.**

19. (*a*) The tearing strengths obtained on the machine-direction specimens and on the cross-direction specimens shall be reported separately in grams. **Report.**

(*b*) The average, the maximum, and the minimum tearing strengths shall be reported for the machine-direction specimens. If possible, similar results shall be reported for the cross-direction specimens.

VII. BURSTING STRENGTH

20. The testing machine shall have a circular flexible diaphragm 6.44 sq. cm. (1 sq. in.) in area. The pressure chamber shall be filled with glycerin or other suitable pressure medium and shall contain no air spaces. The test specimen shall be held in position over the diaphragm in a clamp having a circular hole approximately 6.44 sq. cm. (1 sq. in.) in area so that the diaphragm will force the paper into the hole when pressure is applied under the diaphragm. The pressure exerted on the diaphragm shall be indicated on a dial, graduated to at least 0.23 kg. (0.5 lb.) for papers giving bursting strengths 18 kg. (40 lb.) or under. A dial graduated 0.45 kg. (1 lb.) may be used in testing papers which give higher bursting strengths. The machine may be either hand-operated or power-driven, the latter being preferable. **Apparatus.**

21. If practicable, the specimen shall be so cut from the stock as to permit ten bursting tests on a line across the sheet or roll. **Specimen.**

Procedure. 22. Ten bursts shall be made. The testing machine shall be driven at a uniform speed of 120 r.p.m. until the specimen bursts.

Report. 23. The report shall include the average, the maximum, and the minimum results obtained.

VIII. ABSORPTION

(RISE OF WATER)

Apparatus. 24. A suitable container and support for specimens and scale is shown in Fig. 1.

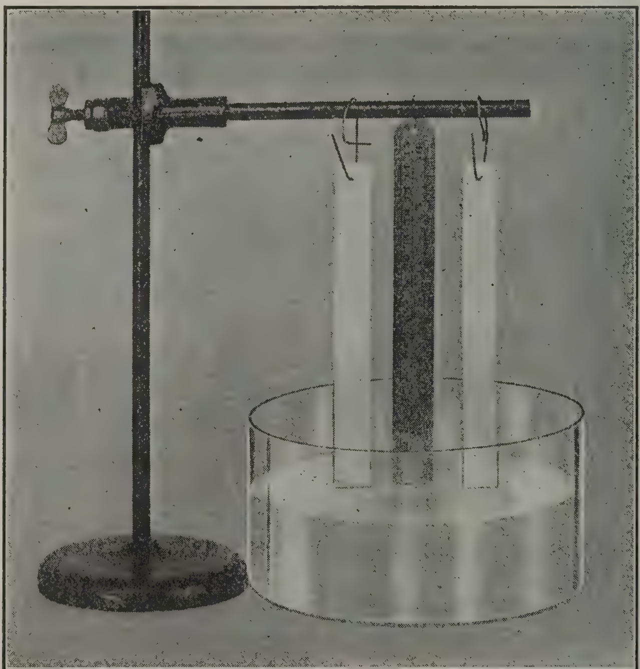

FIG. 1.—Apparatus for Absorption Test.

Specimens. 25. Ten strips 2.5 cm. (1 in.) in width and at least 12.5 cm. (5 in.) in length shall be cut from the samples obtained in accordance with Section 2 (*a*), five being cut parallel with the machine direction of the paper and five being cut parallel with the cross-direction of the paper.

Procedure. 26. The specimens shall be suspended vertically with one end dipping 3.2 mm. (0.125 in.) in distilled water at room temperature.

After five minutes of suspension, the rise of the water in the paper above the water level shall be noted. The rise may be measured by reading directly from the scale the height of absorption.

27. The rise of water shall be reported in millimeters, to the nearest 3 mm. or in inches to the nearest $\frac{1}{8}$ in. **Report.**

IX. MIL WEIGHT PER REAM
(APPARENT DENSITY)

28. A scale, sharp knife, chemical balance and weighing bottle are necessary for the test. **Apparatus.**

29. Duplicate specimens shall be taken from as widely separated portions as possible from each of the samples obtained in accordance with Section 2 (*a*) and conditioned in accordance with Section 3. **Specimens.**

30. (*a*) Each specimen while in the conditioned atmosphere (see Section 3) shall be cut accurately to any convenient size, for example, 232.3 sq. cm. (36 sq. in.) in area, and accurately weighed. Specimens of sufficient size to weigh at least from 3 to 5 g. (0.11 to 0.18 oz.) should be taken. If it is not possible to do the weighing in the conditioned atmosphere, the specimen shall be placed in a weighing bottle and tightly stoppered with a ground glass stopper before being removed from the conditioned atmosphere. The thickness of each specimen shall be determined in accordance with Section 10. **Procedure.**

(*b*) *Mil Weight per Ream of Any Size.*—The mil (or point) weight in pounds per ream of any size may be calculated from the following formula:

$$W = 0.002205 \frac{mSN}{ta}$$

where m = the weight of the specimen expressed in grams;
t = the average thickness of the specimen expressed in mils (thousandths of an inch);
a = the area of the specimen expressed in square inches;
S = the area of one sheet in the basic ream expressed in square inches;
N = the number of sheets in the ream.

NOTE.—0.002205 is contracted from 0.00220462.

NOTE.—The following formulas may be used to calculate the mil weight in pounds per ream for reams of particular sizes, provided a specimen of exactly 232.3 sq. cm. (36 sq. in.) is used:

Ream of 24 by 36 in., 480 sheets.

$$W = 25.40 \frac{m}{t}$$

where m = the weight of specimen expressed in grams;
t = the average thickness of the specimen in mils (thousandths of an inch).

25.40 is contracted from 25.3972224.

Ream of 24 by 36 in., 500 sheets.

$$W = 26.45 \frac{m}{t}$$

where m and t are the same as above and 26.45 is contracted from 26.45544.

NOTE.—The ream weight may be obtained from the above formulas by omitting the thickness "t".

(*c*) *Weight per Unit Volume.*—The weight in pounds per cubic foot (density) may be calculated from the following formula:

$$D = 3810 \frac{m}{ta}$$

Where m = the weight of the specimen expressed in grams.
t = the average thickness of the specimen expressed in mils (thousandths of an inch).
a = the area of the specimen expressed in square inches.

NOTE.—3810 is contracted from 3809.58336.

NOTE.—To convert weight in pounds per cubic foot to mil weight in pounds per ream of 24 by 36 in., 480 sheets, multiply by 0.24; to convert to mil weight in pounds per ream of 24 by 36 in., 500 sheets, multiply by 0.25.

Report. 31. The weight in pounds per cubic foot or the mil weight per ream shall be reported. The size of the ream must be given when the mil weight per ream is reported.

X. AIR RESISTANCE

Apparatus. 32. (*a*) The instrument shall consist of two aluminum open-top cylinders, one of which is inverted and slides into the other which is fixed. The movable cylinder shall be provided with a circular aperture in the closed end and a flat ring clamp for holding the paper specimen across this aperture.

(*b*) The fixed cylinder shall be 25.4 cm. (10 in.) in height and shall have an external diameter of 8.60 cm. (3.38 in.) and an internal diameter of 8.25 cm. (3.25 in.). Four slender bars, each 18.5 cm. (7.3 in.) long, 3 mm. (0.12 in.) wide and approximately 1.5 mm. (0.06 in.) thick shall be mounted vertically and equi-distantly on the inner surface of the fixed cylinder to act as guide tracks for the movable cylinder. The movable cylinder shall be graduated in units of 50 cc. and shall have a total range of 350 cc. It shall be 25.4 cm. (10 in.) high and shall have an external diameter of 7.62 cm. (3.00 in.) and an internal diameter of 7.35 cm. (2.90 in.). It shall weigh, including the flat ring clamp and the two knurled nuts, 567 g. (20 oz.) ± 5 g. (0.18 oz.). The movable cylinder and the flat ring clamp shall have a concentric circular aperture of 6.44 sq. cm. (1.00 sq. in.) in area.

When this aperture is too large for the specimen a similar movable cylinder and flat ring clamp having a concentric circular aperture of 1.61 sq. cm. (0.25 sq. in.) in area shall be used.

NOTE.—Results obtained with apertures of different areas are not directly proportional to the areas of the apertures. The proportionality factor must be determined by experiment.

Specimen. 33. The specimens shall be not less than 3.5 cm. (1.36 in.) nor more than 5.1 cm. (2.0 in.) in width cut from the original samples, taken according to Section 2 (*a*) and shall be as long as the width of the original roll of paper. If the specimens are selected from pads of tape they shall be not less than 2.1 cm. (0.81 in.) nor more than 5.1 cm. (2.0 in.) wide and at least 30 cm. (12 in.) long. There shall be as many specimens as there are original samples.

Procedure. 34. The fixed cylinder shall be placed on a rigid support so that its sides are vertical. Ethylene glycol shall be placed in it to a depth of 12.7 cm. (5 in.). The specimen (one thickness only) shall be secured tightly under the clamp on the movable cylinder completely covering the aperture. The movable cylinder is then floated on the ethylene glycol. The time required for the displacement of a certain amount of air is noted with a stop-watch. If possible, the amount of air displaced should be such that the time of displacement is not less than 20 seconds. The apparatus with its content of ethylene glycol shall be at the temperature of the conditioning room when the readings are taken. The time in seconds required for the displacement of 100 cc. (6.1 cu. in.) through a circular area (one side only) of 6.44 sq. cm. (1 sq. in.) of the paper is known as the air resistance of the paper.

NOTE.—The clamp shall be tested for leakage by substituting a piece of tin foil 0.05 mm. (0.002 in.) in thickness for the paper and testing in the manner described above. When so tested, the leakage shall not exceed the rate of 50 cc. in five hours.

The proper procedure for clamping the specimen or tinfoil is to turn both knurled nuts down onto the clamp simultaneously. If only one nut at a time is turned down, the clamp will not fit flat on the specimen and will consequently have an avoidable leak.

Ethylene glycol is used in preference to distilled water because it does not corrode aluminum whereas distilled water does.

Report. 35. The report shall include (1) the number of seconds required for the displacement of 100 cc. (6.1 cu. in.) of air; (2) the area of paper through which the air was displaced; (3) the thickness of the paper as obtained under Section 11 and (4) the room temperature.

XI. ASH

Apparatus. 36. A suitable crucible such as platinum, nickel or porcelain, a balance sensitive to 0.1 mg., and a desiccator are necessary for the test.

Specimen. 37. Not less than 2 g. of dry, finely-divided paper from the "moisture determination" sample as obtained in accordance with Section 6 shall be used.

Procedure. 38. The sample shall be transferred rapidly from the weighing bottle containing the dried sample into the crucible. The weighing bottle shall immediately be re-stoppered and re-weighed, and the exact weight of the sample obtained by taking the difference in weights of the weighing bottle and the sample. The sample shall be completely ignited in the crucible, preferably in a muffle furnace, all precautions being taken to prevent loss of ash or sample. The residue of incombustible mineral matter (ash) shall be desiccated, cooled and weighed, and the amount computed as a percentage of the weight of the dried sample.

Report. 39. The amount of ash shall be reported as a percentage of the weight of the dry sample (see Section 7 (*a*)) and also as a percentage of the weight of the undried sample (see Section 7 (*b*)).

TENTATIVE METHODS OF TESTING LAMINATED SHEET INSULATING MATERIALS[1]

Serial Designation: D 229 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

1. These tests apply to laminated flat sheet materials intended for use as electrical insulation.[2] **Scope.**

2. In referring to the cutting of the specimens and the application of the load, the following definitions apply: **Definition of Terms.**

Flatwise.—Load applied to the face of the laminations.

Edgewise.—Load applied to the edge of the laminations.

Lengthwise.—In the direction of the length of the sheet.

Crosswise.—In the direction at right angles to the length of the sheet.

NOTE.—When the sheet has the same length and width, one dimension shall arbitrarily be designated as the length, and the other as the width.

I. TENSILE STRENGTH

3. Any standard type of testing machine may be used. The machine shall be accurate to within 1 per cent on the lowest load for which it is used. Jaws which tighten under load, such as wedge grip jaws, shall be used with the specimen properly aligned. **Apparatus.**

4. Tension test specimens shall conform to the dimensions shown in Fig. 1. The surfaces of the specimen shall be smooth and free from scratches. **Specimens.**

5. Five specimens cut lengthwise and five specimens cut crosswise of the sheet shall be tested. **Procedure.**

6. All tests shall be made at normal room temperature of about 20° C. (68° F.). **Temperaature.**

7. The cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced, but shall not exceed 0.05 in. per minute when the machine is running idle. **Speed of Testing Machine.**

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

[2] Tests for shearing strength, compressive strength, moisture absorption, oil absorption and degree of curing are being studied by the committee. Various mechanical tests, particularly applicable to thin laminated sheet materials, are being developed.

8. When it is desired to obtain the modulus of elasticity, a suitable extensometer measuring the elongation on a 2-in. gage length shall be used. The cross-head speed of the testing machine shall not exceed 0.03 in. per minute when the machine is running idle.

9. Strain readings shall be taken by increments of load not exceeding about 1000 lb. per sq. in. The modulus shall be calculated from the total change of length between zero load and stress of 5000 lb. per sq. in.

NOTE.—For some materials the stress strain curve will not form a straight line, but will be a continuous curve from the origin to the breaking load.

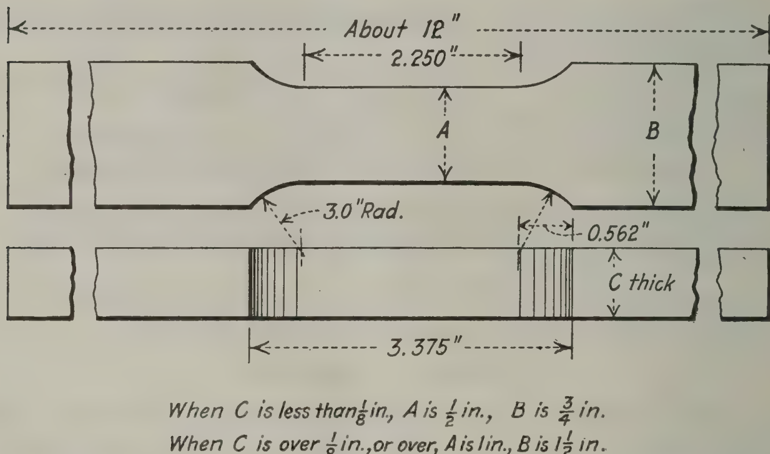

FIG. 1.—Tension Test Specimen for Laminated Sheet Insulating Materials.

Report. 10. The report shall include:

(a) The direction in which the specimen was taken, whether lengthwise or crosswise of the sheet.
(b) The thickness and width of each specimen in inches or centimeters.
(c) The breaking load of each specimen, in pounds or kilograms.
(d) The tensile strength of each specimen, in pounds per square inch or in kilograms per square centimeter.
(e) The location and nature of the fracture.
(f) The modulus of elasticity, if determined.
(g) The room temperature.

II. TRANSVERSE STRENGTH

(*A*) *For Materials ½ in. Thick or Over*[1]

11. (*a*) Any standard type of testing machine may be used. The machine shall have an accuracy of at least 1 per cent of the lowest load for which it is used. **Apparatus.**

(*b*) The distance between points of support shall be 4 in. for specimens ½ in. in thickness and proportionately longer for greater thicknesses. The supports shall have the contact edges rounded to a radius of ⅛ in. The load shall be applied midway between the supports by a pressure piece, the bearing edge of which shall be rounded to a radius of ⅛ in.

12. The test specimen shall be 5 in. in length, ½ in. in width and ½ in. in thickness, for sheets ½ in. in thickness, and for thicker sheets it shall be the full thickness of the sheet with width and length increased in proportion. **Specimen.**

13. The specimens shall be tested as follows: **Procedure.**

(*a*) Five specimens cut lengthwise of sheet, tested flatwise.
(*b*) Five specimens cut lengthwise of sheet, tested edgewise.
(*c*) Five specimens cut crosswise of sheet, tested flatwise.
(*d*) Five specimens cut crosswise of sheet, tested edgewise.

14. All tests shall be made at normal room temperature of about 20° C. (68° F.). **Temperature of Tests.**

15. The cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced, but shall not exceed 0.05 in. per minute when the machine is running idle. **Speed of Testing Machine.**

16. The deflection at the center in inches or millimeters shall be taken at a beam load corresponding to a calculated maximum fiber stress of 10,000 lb. per sq. in. (705 kg. per sq. cm.).

17. The report shall include: **Report.**

(*a*) The directions of cutting and loading of the specimen as specified in Section 13.

(*b*) The thickness and width of each specimen in inches or millimeters.

(*c*) The breaking load of each specimen in pounds or kilograms.

(*d*) The maximum fiber stress in pounds per square inch or kilograms per square centimeter calculated from the formula:

$$S = \frac{3\ Pl}{2\ bd^2}$$

[1] A method of test for thinner materials is being considered.

where S = maximum fiber stress.

P = breaking load in pounds or kilograms;

l = distance between supports in inches or centimeters;

b = width of specimen in inches or centimeters;

d = depth of specimen in inches or centimeters.

(*e*) The room temperature.

(*f*) The deflection at the center in inches or millimeters at a beam load corresponding to a calculated maximum fiber stress of 10,000 lb. per sq. in. (705 kg. per sq. cm.), if determined.

III. DIELECTRIC STRENGTH

18. The dielectric strength shall be determined in accordance with the Tentative Methods of Testing Sheet and Tape Insulating Materials for Dielectric Strength (Serial Designation: D 149 – 24 T) of the American Society for Testing Materials.[1]

IV. PHASE DIFFERENCE (POWER FACTOR) AND DIELECTRIC CONSTANT AT RADIO FREQUENCIES

19. The phase difference and dielectric constant shall be determined in accordance with the Tentative Methods of Testing Electrical Insulating Materials for Phase Difference (Power Factor) and Dielectric Constant at Radio Frequencies (Serial Designation: D 150 – 23 T) of the American Society for Testing Materials.[2]

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[1] See p. 650.
[2] See p. 657.

TENTATIVE SPECIFICATIONS

FOR

WOODEN BOXES, NAILED AND LOCK-CORNER CONSTRUCTION, FOR THE SHIPMENT OF CANNED FOODS.[1]

Serial Designation: D 44 – 20 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1917; Revised, 1920.

Canned Foods Boxes.

Wooden boxes of nailed and lock-corner construction for the shipment of canned foods shall comply with the Tentative General Specifications for Wooden Boxes, Nailed and Lock-Corner Construction (Serial Designation: D 68 – 22 T) of the American Society for Testing Materials,[2] except as otherwise provided in the following table:

Commodity.	Type of Box Construction.	Minimum Thickness of Material, in.[1]			Maximum Gross Weight of Box and Contents, lb.	Permissible Variations in Inside Dimensions of Boxes.
		Ends.	Sides.[2]	Top and Bottom.[2]		
Canned Foods in Metal Cans (Fish, Meats, Fruits, Vegetables and Other Foods)	Nailed	$\frac{5}{8}$	$\frac{5}{16}$	$\frac{5}{16}$	90	Inside dimensions of boxes shall not exceed the exact length and width of contents by more than $\frac{1}{4}$ in., nor the exact depth of contents by more than $\frac{1}{8}$ in.
	Lock-Corner	$\frac{7}{16}$	$\frac{7}{16}$	$\frac{5}{16}$	50	
		$\frac{1}{2}$	$\frac{5}{16}$	$\frac{5}{16}$	50	
		$\frac{5}{8}$	$\frac{5}{16}$	$\frac{5}{16}$	90	

Note 1.—The thicknesses specified apply to boxes of Group I, II and III woods. When woods in Group IV are used the thicknesses may be $\frac{1}{16}$ in. less than those specified.

Note 2.—When one-piece sides and two-piece tops and bottoms of woods of Groups I and II are used, material may be $\frac{1}{32}$ in. thinner than specified. When rotary-cut gum lumber is used in one-piece sides and tops and not more than two-piece bottoms, the thickness may be $\frac{1}{16}$ in. less than specified for woods of Group III, minimum thickness $\frac{1}{4}$ in.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. C. A. Plaskett, Secretary of Committee D-10 on Shipping Containers, Forest Products Laboratory, Madison, Wis.

[2] See p. 704.

TENTATIVE SPECIFICATIONS

FOR

CANNED FOODS BOXES, WIREBOUND CONSTRUCTION.[1]

Serial Designation: D 45 – 17 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1917.

I. MANUFACTURE.

Manufacture. 1. Boxes shall be well manufactured from sound (free from decay or dote), well-seasoned veneer and cleat lumber. Kiln drying by excessively high temperatures or low humidities or below 6 per cent moisture shall be avoided. Veneer shall be free from knot holes, loose or rotten knots greater than 1 in. in diameter. Cleats shall be free from knots and from excessive cross grain. No knots will be permitted which will interfere with proper nailing or stapling. Each side, top, and end shall be of a single piece of veneer; the bottoms shall consist of not more than two pieces, no piece less than 4 in. in width.

Nos. $2\frac{1}{2}$ and 3 Cans. 2. Boxes for two dozen No. $2\frac{1}{2}$, two dozen No. 3, and two dozen extra size No. 3 cans shall be constructed as follows:

(*a*) *Cleats*.—Each end shall be cleated with four cleats not less than $\frac{15}{16}$ by $\frac{11}{16}$ in., or any other size cleats that have equally large cross-section. One end of each cleat shall be provided with a tenon $\frac{1}{4}$ in. thick and $\frac{15}{16}$ in. long and the other end with the corresponding mortise. Cleats may be made of any of the following species of wood:

Red gum	Oak
Black gum	Sycamore
Maple	Ash
Birch	Hickory
Beech	Hackberry
Tupelo	Yellow pine
Elm	

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. C. A. Plaskett, Secretary of Committee D-10 on Shipping Containers, Forest Products Laboratory, Madison, Wis.

(*b*) The sides, top, bottom and ends shall be of $\frac{1}{8}$-in. gum[1] veneer. The box shall be bound with four equally spaced No. 16-gage 0.0625-in. diameter, basic annealed wires. End wires shall be attached to cleats with No. 16-gage by $1\frac{1}{8}$-in. bright, soft bessemer staples driven through the veneer into the center of the width of the cleats. Six staples shall be driven into each top and bottom cleat and four into each side cleat. Interior wires shall be attached to the veneer by No. 18-gage, bright, soft bessemer staples driven through and clinched; six staples connecting each wire to top and bottom and four to each side.

(*c*) The ends shall be attached to each side cleat by four No. 18-gage by $\frac{13}{16}$-in. staples equally spaced, driven into the center of the cleat and at an angle of 45 to 60 deg. with the grain of the end; or by four nails $\frac{7}{8}$-in. by 15-gage by $\frac{1}{4}$-in. head, driven into the center of the cleat and equally spaced.

Nos. 1 and 2 Cans.

3. Boxes for two dozen No. 1, four dozen No. 1, and two dozen No. 2 cans shall be constructed as follows:

These boxes shall be the same as those for No. 3 cans, except that wires shall be stapled to the tops and bottoms with four staples to each wire and to the sides with three staples to each wire.

[1] Specifications for boxes of other kinds of veneer and with cleats of other species than those listed have been withheld pending tests.

TENTATIVE GENERAL SPECIFICATIONS

FOR

WOODEN BOXES, NAILED AND LOCK-CORNER CONSTRUCTION[1]

Serial Designation: D 68 – 22 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1920; REVISED, 1922

Types of Boxes Covered.

1. These specifications are intended to apply to wooden boxes of nailed and lock-corner construction, and are directly applicable to the styles of boxes illustrated in Fig. 1.

I. MATERIAL

Seasoning.

2. The parts shall be made from thoroughly seasoned lumber. Under average conditions, thoroughly seasoned lumber has a moisture content of 12 to 18 per cent of the weight of the wood after oven drying to a constant weight.

Defects.

3. All defects[2] in the lumber that materially lessen the strength of the part, expose the contents to damage, or interfere with the prescribed nailing, shall be eliminated.

Manufacture.

4. Ends, sides, tops, bottoms and other parts of a box shall be well manufactured and cut true to size.

II. GROUPING OF WOODS

Grouping of Woods.

5. The principal woods used for boxes are classed for the purpose of specifications into four groups, as follows:

GROUP I

White pine	Willow
Norway pine	Noble fir
Aspen (Popple)	Magnolia
Spruce	Buckeye
Western (yellow) pine	White fir

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. C. A. Plaskett, Secretary of Committee D-10 on Shipping Containers, Forest Products Laboratory, Madison, Wis.

[2] The committee recognizes that the limitations on defects are not sufficiently definitive. This phase is being made the subject of careful study with the object of recommending more definite requirements.

Cottonwood	Cedar
Yellow poplar	Redwood
Balsam fir	Butternut
Chestnut	Cucumber
Sugar pine	Alpine fir
Cypress	Lodgepole pine
Basswood	Jack pine

GROUP II.

Southern yellow pine	Douglas fir
Hemlock	Larch (Tamarack)
North Carolina pine	

GROUP III.

White elm	Black ash
Red gum	Black gum
Sycamore	Tupelo
Pumpkin ash	Maple, soft or silver

GROUP IV.

Hard maple	Birch
Beech	Rock elm
Oak	White ash
Hackberry	Hickory

III. DIMENSIONS OF PARTS.

Thickness of Parts.

6. (*a*) The thicknesses called for in specifications for boxes of any given commodity will, unless otherwise specified, be understood as applying to woods of Groups I and II.

(*b*) When the thicknesses of material in the following table are specified (for Groups I and II woods), woods of Groups III and IV may be used with the following permissible reduction in thickness:

Specified Thickness of Parts (Groups I and II).	Allowable Reduction in Thickness for Groups III and IV.
$\frac{1}{4}$ to $\frac{1}{2}$ in., incl.	$\frac{1}{16}$ in.
Over $\frac{1}{2}$ to 1 in., incl.	$\frac{1}{8}$ in.
1 to 2 in., incl.	$\frac{1}{4}$ in.

Occasional Variation in Thickness of Parts.

7. The occasional variation below the thickness of parts specified in Section 6 shall not exceed one-eighth of the thickness of the part.

Number of Pieces.

8. (*a*) The maximum number of pieces allowed in any side, top, bottom or end of a box shall be as follows:

Width of Part.	Maximum Number of Pieces.
4 in. and under	1
Over 4 to 7 in., incl.	2
" 7 to 10 in., incl.	3

Over 10 in. shall average not more than one piece for each 3 in. of width and no piece shall be less than $2\frac{1}{2}$ in. in width. Boards matched and fastened as provided in the following section may be not less than $1\frac{1}{2}$ in. in width at either end.

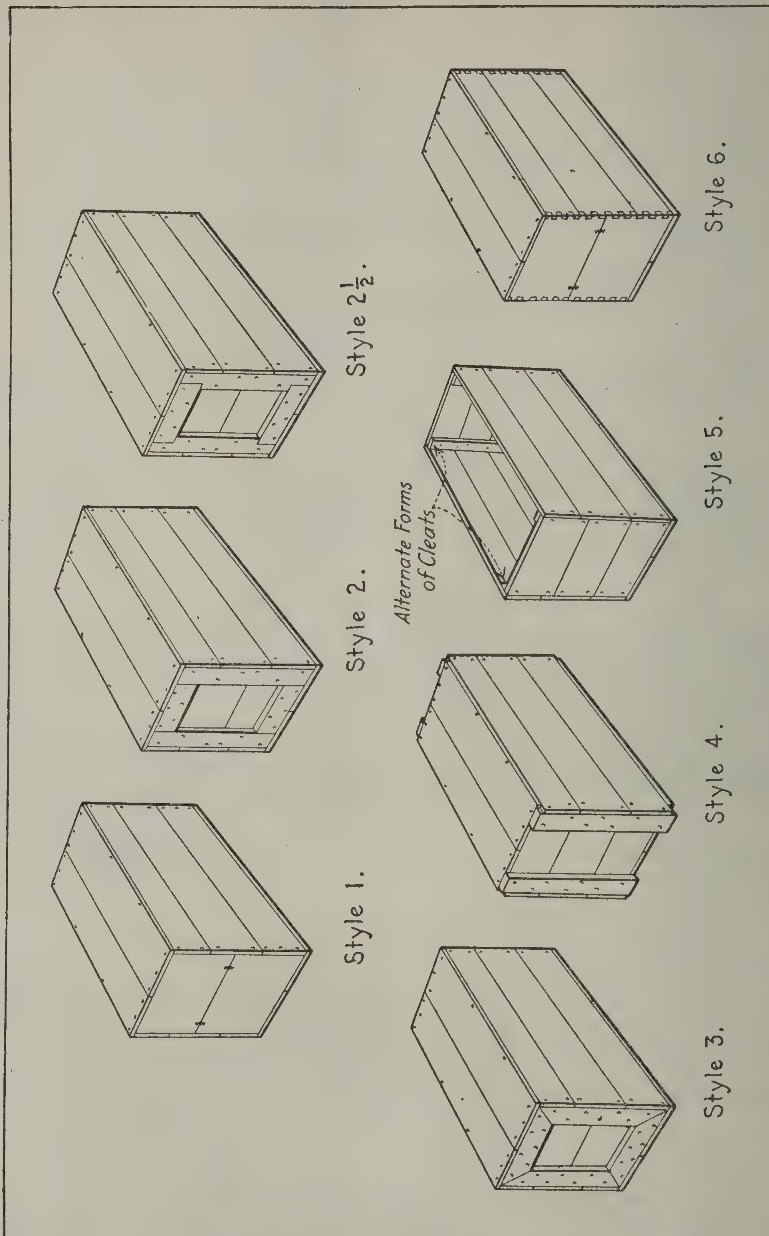

FIG. 1.—Styles of Wooden Boxes, Nailed and Lock-Corner Construction.

(*b*) Two or more pieces $\frac{1}{2}$ in. or over in thickness, fastened as follows shall be considered one piece in sides, tops and bottoms: butt-jointed or matched and fastened with two or more corrugated fasteners $1\frac{1}{8}$ in. in length and of sufficient width to penetrate approximately three-fourths the thickness of the material.

IV. MANUFACTURE OF BOXES.

9. The outside surface of boxes shall be sufficiently smooth to permit of legible marking. When surfacing of boards two sides is necessary for the protection of the commodities in transportation, the material may be $\frac{1}{32}$ in. thinner than the minimum specified. **Surfacing of Boxes.**

10. (*a*) Ends $\frac{7}{8}$ in. or less in thickness, if made of two or more pieces, shall be: (1) butt-jointed or matched, and fastened with two or more corrugated fasteners; or (2) cleated. **Joining.**

(*b*) The sizes of corrugated fasteners used shall be as follows:

Thickness of End, in.	Size of Fastener, in.
$\frac{3}{8}$	$\frac{1}{4}$ by $1\frac{1}{8}$
$\frac{7}{16}$, $\frac{1}{2}$ and $\frac{9}{16}$	$\frac{3}{8}$ by $1\frac{1}{8}$
$\frac{5}{8}$, $\frac{11}{16}$ and $\frac{3}{4}$	$\frac{1}{2}$ by $1\frac{1}{8}$
$\frac{13}{16}$ and $\frac{7}{8}$	$\frac{5}{8}$ by $1\frac{1}{8}$

(*c*) The number of fasteners used shall be as follows:

Length of End.	Number of Fasteners.
16 in. and under	2
Over 16 to 24 in.	3
" 24 " 36 "	4

(*d*) When three or more fasteners are required, they shall be driven alternately from opposite sides of the end.

(*e*) Two or more pieces Linderman jointed shall be considered one piece.

Schedule of Nailing.

11. (*a*) All nails, except as specified in Paragraph (*b*), shall be standard cement-coated box nails of the following length and gage (Washburn and Moen or American Steel and Wire Co. gage): **Kind of Nails.**

Size of Nails, "penny."	Length.	Gage No.
2	1 in.	16
3	$1\frac{1}{8}$ "	$15\frac{1}{2}$
4	$1\frac{3}{8}$ "	14
5	$1\frac{5}{8}$ "	$13\frac{1}{2}$
6	$1\frac{7}{8}$ "	13
7	$2\frac{1}{8}$ "	$12\frac{1}{2}$
8	$2\frac{3}{8}$ "	$11\frac{1}{2}$
9	$2\frac{5}{8}$ "	$11\frac{1}{2}$

(*b*) If other than cement-coated nails are used, 25 per cent more nails shall be driven than specified for cement-coated nails. Plain nails driven through and clinched may be used for cleating.

Size of Nails. 12. The size of nails to be used is determined by the species and thickness of the material in which the points of the nails are held, and shall be in accordance with the following table:

SIZE OF CEMENT-COATED NAILS FOR DIFFERENT SPECIES AND THICKNESS OF MATERIAL.

(The sizes of nails are given in "penny".)

Species of Wood Holding Nails.	Thickness of Ends or Cleats to Which Sides, Tops and Bottoms are Nailed, in.								Thickness of Sides to Which Top and Bottom are Nailed, in.		
	$\frac{3}{8}$ or less	$\frac{7}{16}$	$\frac{1}{2}$	$\frac{9}{16}$	$\frac{5}{8}$	$\frac{11}{16}$ or $\frac{3}{4}$	$\frac{13}{16}$	$\frac{7}{8}$	Less than $\frac{1}{2}$	$\frac{1}{2}$ to $\frac{9}{16}$	$\frac{5}{8}$ to $\frac{7}{8}$
Group I woods	4	5	5	6	7	8	8	9	4	6	7
Group II woods	4	4	5	5	6	7	7	8	4	5	6
Group III woods	3	4	4	5	5	6	7	7	3	4	5
Group IV woods	3	3	4	4	4	5	6	7	3	4	5

NOTE.—If the designated penny of nail is not available, the next lower penny shall be used, and the nails spaced proportionately closer.

Spacing and Number of Nails. 13. (*a*) The spacing of nails holding the side, top or bottom to the ends of a box shall be as follows:

SIZE OF NAILS, "PENNY."	SPACE WHEN DRIVEN INTO SIDE GRAIN OF END.	SPACE WHEN DRIVEN INTO END GRAIN OF END.
6 or less	2 in.	$1\frac{3}{4}$ in.
7	$2\frac{1}{4}$ "	2 "
8	$2\frac{1}{2}$ "	$2\frac{1}{4}$ "
9	$2\frac{3}{4}$ "	$2\frac{1}{2}$ "
10	3 "	$2\frac{3}{4}$ "

(In order to ascertain the number of nails to be used, divide the width of the side, top or bottom (or length of cleat) by the spacing specified for the size of nails to be used. Fractions in the result greater than $\frac{1}{4}$ if the points of nails are to be held in the end grain, and greater than $\frac{1}{2}$ if the points of nails are to be held in the side grain, will be considered as a whole number.)

(*b*) When side nailing is used the spacing of nails holding top and bottom to sides shall be 6 to 8 in. When the material in the sides is less than $\frac{1}{2}$ in. in thickness, side nails shall not be used, unless otherwise specified.

(*c*) No boards shall have less than two nails at each nailing end, except boards less than $2\frac{1}{2}$ in. in width, which are butt-jointed or matched and fastened with corrugated fasteners as specified in Section 8 (*b*) may have only one nail. When cleats of the thickness of

the ends are used, the nails shall be driven in the ends and cleats alternately.

(*d*) When box ends are cleated, the spacing of nails holding cleats and ends together shall be the same as the spacing of the nails holding the adjacent side, top, or bottom to the end. Each piece of the end shall be nailed to each cleat with not less than two nails, except that end boards less than $2\frac{1}{2}$ in. in width which are butt-jointed or matched and fastened with corrugated fasteners as provided for in Section 8 (*b*) may have only one nail.

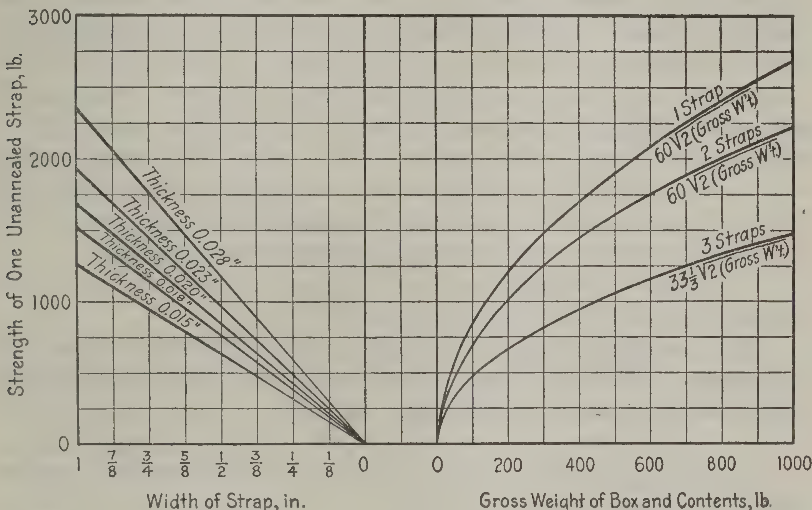

FIG. 2.—Chart for Determining Size of Strap.

(*e*) Nails attaching cleats to ends shall be of sufficient length to penetrate both thicknesses of lumber and allow for clinching. Either cement-coated or plain nails may be used.

(*f*) When cleats are thicker than the ends, nails shall be driven through the end boards into the cleats and clinched.

Driving of Nails.

14. Nails shall be driven flush.

(Over-driving of nails materially weakens the container.)

V. STRAPPING OF BOXES.

Strapping.

15. When boxes are specified to be strapped with flat metal straps, either (1) unannealed straps without nails or (2) annealed or unannealed straps nailed on, shall be used, subject to the following requirements:

(*a*) When the straps are nailed on, they shall be placed around the ends of the box and held in position by the same size nails spaced twice the distance specified for holding the box parts together.

(*b*) Metal fasteners used to hold the ends of nailless straps together shall have not less than 75 per cent of the tensile strength of the strap.

(*c*) All straps shall be drawn tight so as to sink into the corners of the box.

(*d*) When two or three nailless straps are used, one shall be placed approximately one-sixth of the length of the box from each end.

(*e*) Fig. 2 gives the size of straps to be used on various gross weights of boxes. Starting with the weight of box and contents, move directly upward to the curve for the number of straps decided upon; then move to the left to an intersection with the line representing the thickness of straps to be used. The first vertical line at or beyond this point of intersection represents the width of strap required; or, move to the left, as above, to the vertical line representing the width of strap decided upon and the first diagonal line above this point of intersection represent the thickness of strap required.

(*f*) Material wider than $\frac{3}{4}$ in. or thicker than 0.023 in. is not ordinarily used for nailless strapping.

Thickness of Parts of Strapped Boxes.

16. The thicknesses specified for sides, tops, and bottoms of unstrapped nailed boxes may be reduced to the thickness indicated below, when metal straps are used:

Specified Thickness of Sides, Tops, and Bottoms of Unstrapped Boxes, in.	Thickness of Sides, Tops, and Bottoms of Strapped Boxes, in.	
	One Strap.	Two or More Straps.
$\frac{7}{8}$	$\frac{5}{8}$	$\frac{1}{2}$
$\frac{13}{16}$	$\frac{5}{8}$	$\frac{1}{2}$
$\frac{5}{8}$	$\frac{1}{2}$	$\frac{3}{8}$
$\frac{9}{16}$	$\frac{7}{16}$	$\frac{5}{16}$
$\frac{1}{2}$	$\frac{3}{8}$	$\frac{5}{16}$
$\frac{7}{16}$	$\frac{5}{16}$	$\frac{1}{4}$
$\frac{3}{8}$	$\frac{5}{16}$	$\frac{1}{4}$

TENTATIVE GENERAL SPECIFICATIONS
FOR
4-ONE BOXES AND SIMILAR TYPE BOXES[1]

Serial Designation: D 118 – 21 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921

1. These specifications cover three styles of 4-One and similar type boxes as follows: **Material Covered.**

(*a*) General form.

(*b*) Boxes with wedglock ends.

(*c*) Boxes with detached tops.

GENERAL FORM.

2. (*a*) The boxes knocked down shall consist of separate sections forming top, sides, and bottom, connected only by continuous steel binding wires; and of separate ends.

(*b*) Each of the separate sections forming the sides, top and bottom shall consist of cleats, thin boards, wire and staples.

(*c*) The separate sections shall be separated such a distance from each other that the wires shall be in tension at the corners when the sections are folded.

I. GROUPING OF WOODS.

3. For the purposes of these specifications, box lumber shall be classed into four groups as follows: **Grouping of Woods.**

GROUP I.

Alpine fir	Cottonwood	Redwood
Aspen	Cucumber	Spruce
Balsam fir	Cypress	Sugar pine
Basswood	Jack pine	Western (yellow) pine
Buckeye	Lodgepole pine	White fir
Butternut	Magnolia	White pine
Cedar	Noble fir	Willow
Chestnut	Norway pine	Yellow poplar

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. C. A. Plaskett, Secretary of Committee D-10 on Shipping Containers, Forest Products Laboratory, Madison, Wis.

GROUP II.

Douglas fir	Larch (tamarack)	Virginia and Carolina pine
Hemlock	Southern yellow pine	

GROUP III.

Black ash	Red gum	Sycamore
Black gum	Red gum sapwood (commonly called sap gum)	Tupelo
Maple, soft or silver		White Elm
Pumpkin ash		

GROUP IV.

Beech	Hickory	Oak
Birch	Maple, hard	Rock elm
Hackberry		White ash

II. MATERIALS.

Cleats. 4. (*a*) Each cleat shall be sound, free from knots and from cross grain which runs across it within a distance equal to one-half its length.

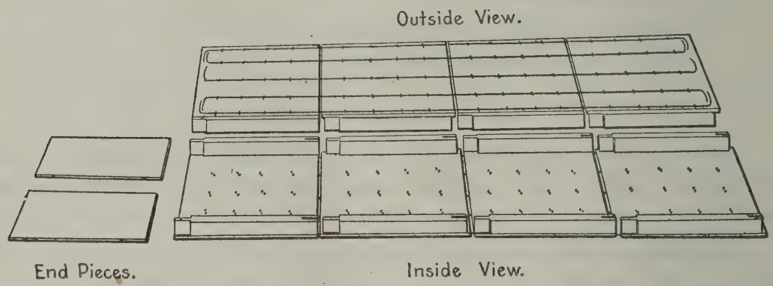

FIG. 1.

(*b*) Cleats shall be not less than $\frac{3}{4}$ in. thick (parallel to the length of the box) and not less than $\frac{7}{8}$ in. in width.

Thin Boards. 5. (*a*) The thin boards shall be sound (free from decay and dote), well seasoned and cut so that adjacent faces of boxes will be at right angles to each other. All defects that would materially lessen the strength, expose the contents of the box to damage, or interfere with the proper assembly of the box shall be eliminated.

(*b*) When the thickness of thin boards as specified is less than $\frac{3}{16}$ in., thin boards made of woods of Groups III and IV may be $\frac{1}{32}$ in. less than the specified thickness, except that the minimum thickness of thin boards of any kind of wood shall be $\frac{1}{8}$ in.

(*c*) The variation in thickness of thin boards below the thickness specified shall be not more than $\frac{1}{8}$ of the thickness of the thin board and this variation below the specified thickness shall not extend over more than 10 per cent of the face of that particular board.

(*d*) Thin boards less than $2\frac{1}{2}$ in. in width at either end shall not be used.

6. The binding wires shall be annealed steel wire of not less than No. 16 gage. **Binding Wire.**

7. (*a*) The staples on end wires shall be not less than No. 16 gage by $1\frac{1}{8}$ in. long. **Staples.**

(*b*) Staples on intermediate wires shall be not less than No. 18 gage by $\frac{7}{16}$ in. long.

III. ASSEMBLING.

8. (*a*) The staples on end wires shall be driven home astride the binding wires, through the thin boards into the cleats, and anchored in the cleats. **Driving of Staples.**

(*b*) The staples on intermediate wires shall be driven astride the binding wires, through the thin boards and firmly clinched.

(*c*) The space between staples shall be the average distance between centers of staples astride each binding wire in each section and this space shall be not more than $2\frac{1}{2}$ in. except as specified in Paragraph (*d*).

(*d*) When cleats are made of woods of Groups III and IV, the space between staples may be $\frac{1}{4}$ in. greater than that specified.

(*e*) There shall be not less than two staples driven astride each wire and into each thin board.

(*f*) The staples nearest the corners shall be not more than $1\frac{3}{4}$ in. from the corner to which it is adjacent.

9. Each end of the box shall be securely fastened on the inside of the side cleats with staples not less than No. 16 gage by $\frac{13}{16}$ in. long, or with cement-coated nails of not less than two-penny size. There shall be no space exceeding $2\frac{1}{2}$ in. on any side cleat into which no staple or nail holding the end in place has been driven and there shall be a staple or nail within $1\frac{1}{2}$ in. of each end of each side cleat. Staples or nails shall be driven home. **Fastening of Ends.**

10. At each corner one section shall overlap its adjacent section at right angles and the wire shall be in tension, giving a square, tight corner. **Box Corners.**

11. The cover shall be closed tightly and the ends of each binding wire twisted tightly together. The twisted portion of each wire shall be not less than $\frac{1}{2}$ in. long. The rough ends of the wires shall be removed and the twisted portion driven flat against the side, parallel with the binding wire. **Fastening Cover.**

12. Nothing herein contained shall be construed as prohibiting the use of boxes constructed of thicker thin-boards, additional or **Use of Heavier Material.**

heavier wires, heavier cleats, longer staples, or with closer spacing of staples.

BOXES WITH WEDGLOCK ENDS.

Boxes with Wedglock Ends. 13. These boxes shall consist of sides, top and bottom and one end made in accordance with Sections 2 to 12, inclusive, and of one wedglock end.

Wedglock Ends. 14. The wedglock ends shall consist of the following:

(*a*) One or more thin boards whose thickness is not less than that of the thin boards in the other portions of the box and whose combined width is $\frac{1}{8}$ in. less than the shortest distance between the top and bottom cleats of the box and whose length is the same as the inside width of the box less the width of one of the side cleats.

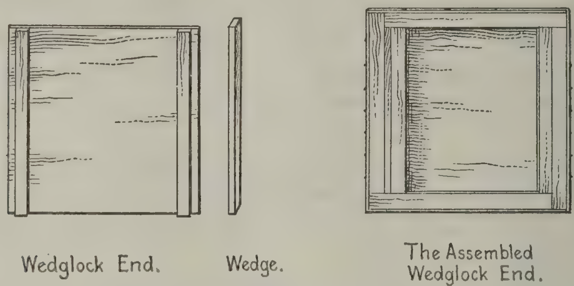

FIG. 2.—Wedglock End.

(*b*) Two battens of the same thickness and width as the cleats in the box and whose length is $\frac{1}{8}$ in. less than the shortest distance between the top and bottom cleats.

(*c*) One wedge of the same thickness and length as the battens and whose width is one-half that of the battens.

Assembling of End. 15. The battens shall be attached across the grain of the thin boards, one batten its own width from one end and the other batten half its width from the other end of the thin-boards, with staples not less than No. 16 gage by $\frac{13}{16}$ in. long or with nails not less than two-penny in size. There shall be no space exceeding 2 in. on any batten into which no staple or nail holding the thin-boards to the batten has been driven and there shall be a staple or nail within $1\frac{1}{2}$ in. of each end of each batten. Staples or nails shall be driven home.

NOTE.—In making up the box, the wedglock end is left out so that the box may be filled from the end. The other end of the box is fastened in place and the box made up as specified in Sections 9 to 11. The wedglock end is not fastened in place until the box is closed.

Boxes with wedglock ends are closed as follows: The wedglock end is inserted. The wedge is inserted. The batten that rests against the wedge is fastened to the wedge with one four-penny nail driven through the middle of the batten into the wedge. The other batten that rests against the cleat is fastened to that cleat with four-penny nails driven through the batten into the cleat. Nail centers shall be not more than 4 in. apart and there shall be a nail within at least 2 in. of each end of this batten.

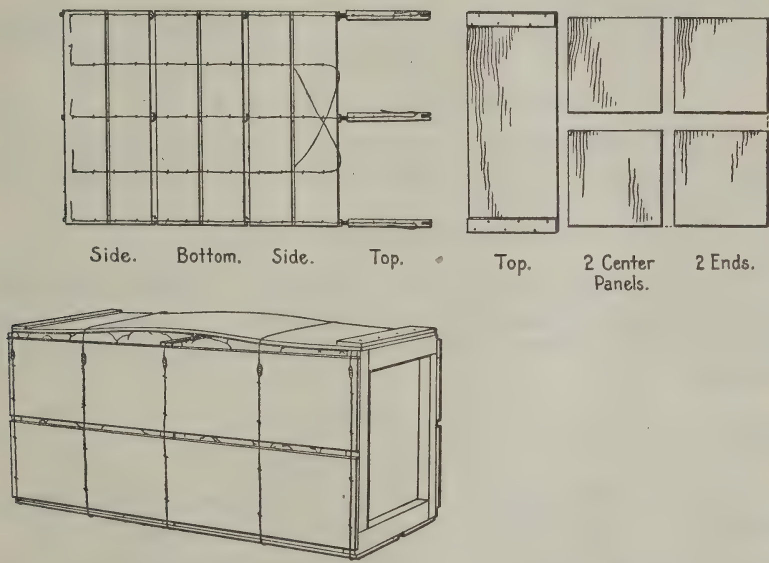

FIG. 3.

BOXES WITH DETACHED TOPS.

16. These boxes shall consist of sides, bottom and ends made in accordance with Sections 2 to 12, inclusive, and a detached top. **Boxes with Detached Tops.**

17. The detached top shall consist of thin boards assembled as shown in Fig. 3. **Detached Top.**

18. In assembling the box, the top cleats to which the binding wires have been stapled shall be put in position on the side cleats and the ends of each wire stapled to the cleats twisted tightly together. **Assembling.**

The detached top shall be nailed to the end cleats with cement-coated box nails spaced not more than $2\frac{1}{2}$ in. apart. The wires not stapled to the cleats shall be brought over the detached top and the ends of each wire twisted tightly together.

TENTATIVE SPECIFICATIONS

FOR

INSULATED WIRE AND CABLE: 30-PER-CENT HEVEA RUBBER.[1]

Serial Designation: D 27 – 21 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1916; REVISED, 1921.

Scope. 1. These specifications cover conductors, rubber insulation, rubber-filled cloth tape and braid. The design and factor of safety depend upon the service conditions and shall be at the option of the purchaser.

(*A*) CONDUCTOR.

I. MANUFACTURE.

Material. 2. The conductor shall be of soft annealed copper, properly tinned, and have the properties and characteristics herein required.

Strand. 3. Each individual wire of a stranded conductor shall be considered separately and shall be designated as a strand.

Shape. 4. Each solid conductor and each strand shall be round and reasonably free from imperfections.

Stranding. 5. The stranding shall be concentric and unless otherwise specified shall conform to either Table I or Table II.

Density. 6. For the purpose of calculating weights, cross-sections, etc., in conductivity determinations, the density of copper shall be taken as 8.89. (See Appendix.)

II. PERMISSIBLE VARIATIONS IN DIMENSIONS.

Diameter and Area. 7. (*a*) *Permissible Variation.*—When the diameter of solid conductors and strands is specified, the permissible variation from the specified value shall not exceed 1 per cent under or 2 per cent over for wire 0.02 in. in diameter and larger, and 0.1 mil under for wire less than 0.02 in. in diameter.

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[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. E. H. Grafton, Secretary of Committee D-11 on Rubber Products, Quaker City Rubber Co., Wissinoming, Philadelphia, Pa.

TABLE I.—STANDARD STRANDING FOR CABLE.
(AMERICAN INSTITUTE OF ELECTRICAL ENGINEERS.)

Size. (See Note 1.)		Number of Wires (See Note 2).	
	Sq. mm.	A Bare, insulated or weatherproof cables for aerial use.	B Insulated cables for other than aerial use.
2.0 cir. in.	1013	91	127
1.5 "	760	61	91
1.0 "	507	61	61
0.6 "	304	37	61
0.5 "	253	37	37
0.4 "	203	19	37
0000 A.w.g.	107	19 or 7 (See note 3)	19
00 "	67.4	7	19
2 "	33.6	7	7
7 and smaller	10.5	...	7

NOTE 1.—For intermediate sizes, use stranding for next larger size. A circular inch equals 1 000 000 circular mils.

NOTE 2.—Conductors of 0000 A.w.g. and smaller are often made solid and this table of stranding should not be interpreted as excluding this practice.

NOTE 3.—Class A cables, sizes 0000 and 000 A.w.g., are usually made of 7 strands when bare and 19 strands when insulated or weather proof.

TABLE II.—PROPOSED STANDARD STRANDING FOR CABLES.[1]
(AMERICAN INSTITUTE OF ELECTRICAL ENGINEERS.)

Strands.			Total Nominal Cross-Section, circular mils.	Total Diameter, in.
Number and Size. (See Note.)	Individual Strands.			
	Nominal diameter, mils.	Nominal Circular mils.		
127 No. 8	128.5	16 510	2 097 000	1.671
127 No. 9	114.4	13 090	1 662 000	1.487
91 No. 8	128.5	16 510	1 502 000	1.414
91 No. 9	114.4	13 090	1 191 000	1.258
61 No. 8	128.5	16 510	1 007 000	1.157
61 – 121 mils	121.0	14 641	893 100	1.089
61 No. 9	114.4	13 090	798 500	1.030
61 – 107 mils	107.0	11 449	698 400	0.963
61 No. 10	101.9	10 380	633 200	0.917
37 – 116 mils	116.0	13 456	497 900	0.812
37 No. 10	101.9	10 380	384 100	0.713
37 – 97 mils	97.0	9 409	348 100	0.679
37 No. 11	90.74	8 234	304 700	0.635
19 No. 9	114.4	13 090	248 700	0.572
19 – 107 mils	107.0	11 449	217 500	0.535
19 No. 11	90.74	8 234	156 400	0.454
19 No. 12	80.81	6 530	124 100	0.404
19 No. 13	71.96	5 178	98 380	0.360
19 No. 14	64.08	4 107	78 030	0.320
7 No. 10	101.9	10 380	72 660	0.306
7 No. 11	90.74	8 234	57 640	0.272
7 No. 12	80.81	6 530	45 710	0.242
7 No. 14	64.08	4 107	28 750	0.192
7 No. 16	50.82	2 583	18 080	0.152
7 No. 18	40.30	1 624	11 370	0.121
7 No. 20	31.96	1 022	7 154	0.096
7 No. 22	25.35	642.4	4 497	0.076
7 No. 24	20.10	404.0	2 828	0.060

NOTE.—Nominal diameters and circular mils of the individual strands are taken from Table VI, *Circular No. 31* of the Bureau of Standards.

Sizes are expressed as A.w.g. gage numbers except where diameters are given in mils.

[1] This table is proposed by the A. I. E. E., but the following note appears at the head of the table: "This table is offered for consideration but will not be recommended for final adoption until ratified by other societies interested."

NOTE.—These limits differ from those in the Standard Specifications for Tinned Soft or Annealed Copper Wire for Rubber Insulation (Serial Designation: B 33) of the American Society for Testing Materials[1] because of the additional manipulation to which the wire is subjected subsequent to the stage where Specifications B 33 apply.

When the area of cross-section of cables is specified, the permissible variation shall not exceed 1 per cent under the specified value.

(*b*) *Method of Gaging Diameter.*—When wire is submitted in coils, each coil shall be gaged in three places, one near each end and one approximately at the middle. When wire is submitted on spools or reels, approximately 12 ft. of the wire shall be reeled off and the wire then gaged in six places between the second foot and the twelfth foot from the end.

(*c*) *Calculation of Area.*—The area of cross-section of wire shall be calculated from the average of the measurements of the diameter made in accordance with Paragraph (*b*). The area of cross-section of cable shall be considered to be the sum of the cross-sectional areas of its component wires, when laid out straight and measured perpendicular to their axes.

TABLE III.—TENSILE PROPERTIES OF CONDUCTORS.

Diameter, in.	Maximum Tensile Strength, lb. per sq. in.	Minimum Elongation in 10 in., per cent.
0.460 to 0.290	36,000	30
0.289 " 0.103	37,000	25
0.102 " 0.021	38,500	20
0.020 " 0.012	39,000	15
0.011 " 0.003	40,000	10

(*d*) *Rejection.*—A coil or reel shall be rejected if the average diameter obtained from the measurements made in accordance with Paragraph (*b*), or the area calculated in accordance with Paragraph (*c*), is not within the limits specified in Paragraph (*a*).

A coil or reel shall be rejected if any individual measurement of diameter or of area of cross-section is not within twice the limits of Paragraph (*a*).

III. PHYSICAL PROPERTIES AND TESTS.

Tensile Properties.

8. (*a*) Each solid conductor and each strand previous to stranding shall be so drawn and annealed that after tinning it shall conform to the values in Table III.

After stranding, the tensile strength of each strand shall not exceed that specified in Table III by more than 5 per cent and the minimum elongation in 10 in. shall not be less than that specified in Table III by more than 5 per cent.

[1] 1924 Book of A.S.T.M. Standards.

(*b*) For nominal diameters between listed sizes the requirements shall be those of the next larger sizes indicated in Table III.

Tension Tests.

9. (*a*) *Method of Test.*—Tension tests shall be made on fair samples and the elongation shall be determined as the permanent increase in length, due to the breaking of the wire in tension, measured between bench marks originally 10 in. apart. The specimen shall break between the bench marks and not closer than 1 in. to either mark.

(*b*) *Number of Tests.*—Samples shall be taken in accordance with Section 18.

(*c*) *Retests and Rejections.*—If upon testing a sample from a coil, reel or spool of wire, the results are found to be above the stated value for tensile strength or below the stated value for elongation, tests upon two additional samples will be made and the average of the three tests shall determine acceptance or rejection.

Conductivity.

10. The conductivity of the conductors after tinning shall be not less than the following values:

DIAMETER, IN.	CONDUCTIVITY, PER CENT.
0.460 to 0.290	97.65
0.289 " 0.103	97.15
0.102 " 0.021	96.15
0.020 " 0.012	94.15
0.011 " 0.003	93.15

NOTE.—The above values for conductivity are based on the International Standard for Annealed Copper Wire. (See Appendix.)

Tinning Test.

11. (*a*) The continuity of the tin coating of each sample before stranding or insulating shall be determined by the Sodium Polysulfide test which shall be made in accordance with the requirements of Sections 4 to 10 of the Standard Specifications for Tinned Soft or Annealed Copper Wire for Rubber Insulation (Serial Designation: B 33) of the American Society for Testing Materials.[1]

(*b*) *Number of Tests.*—Samples shall be taken in accordance with Section 18.

(*c*) *Retests and Rejections.*—If the specimens tested in accordance with Paragraph (*a*) shows any signs of blackening, two more specimens shall be tested. If one of these two additional specimens shows any blackening, that coil or reel shall be rejected. If both specimens are free from any signs of blackening, the coil or reel shall be accepted. If more than 10 per cent of the samples in the entire order fail, all of the wire shall be rejected. If 10 per cent or less of the samples in the entire lot fail, each coil, reel or length may be tested and accepted or rejected upon the results of the individual tests.

[1] 1924 Book of A.S.T.M. Standards.

(*B*) INSULATION.

Workmanship. 12. (*a*) The insulation shall be a properly vulcanized rubber compound, homogeneous in character, tough, elastic and applied concentrically about the conductor and shall fit tightly thereto. When the insulation is applied in more than one layer, adjacent layers shall vulcanize into a homogeneous mass. Tape may be applied over the insulation before vulcanizing but such tape, if it does not meet the tape specifications given in Sections 24-27 inclusive, shall be additional to that required.

(*b*) If exigencies of manufacture require repairs or joints in the insulation, the work shall be done in such manner that the repaired part or joint, and all parts affected by it, shall be as strong and durable electrically, as the remainder of the insulation.

TABLE IV.—THICKNESS OF INSULATION.
(Thickness in sixty-fourths of an inch.)
(AMERICAN INSTITUTE OF ELECTRICAL ENGINEERS.)

Size of Conductor.		Working Pressure, Volts Alternating.										
A.w.g. or Cir. Mils.	Sq. mm.	600 or less	1,500	2,500	3,500	5,000	6,000	7,000	8,000	9,000	10,000	11,000
14 - 8	2.08 - 8.37	3	6	8	10	12	14	16	18	20	22	24
7 - 2	10.6 - 33.6	4	7	9	10	12	14	16	18	20	22	24
1 - 0000	42.4 - 107	5	8	10	10	12	14	16	18	20	22	24
250,000 - 500,000 C.M.	127 - 253	6	9	10	11	12	14	16	18	20	22	24
550,000 - 1,000,000 C.M.	279 - 507	7	10	10	12	12	14	16	18	20	22	24
1,250,000 - 2,000,000 C.M.	633 - 1013	8	10	10	12	14	16	18	18	20	22	24

NOTE.—In multiple conductor cables, the thickness of insulation on each conductor shall be based on the highest r. m. s. voltage between the conductor and the outside of this insulation.

The above table is based upon alternating voltages of commercial frequencies. For voltages over 600, the insulation thickness for direct-current cable has not been established. For intermediate sizes the insulation thickness should be the same as for the next larger sizes.

IV. THICKNESS.

Thickness. 13. (*a*) The thickness of the insulation shall conform to Table IV.

(*b*) *Permissible Variation.*—The average thickness of the insulation shall be not less than that specified. The thickness of the insulation at the thinnest part shall be not less than 90 per cent of the specified thickness, but in no case shall it be more than 0.03125 ($\frac{1}{32}$) in. less than the specified thickness.

(*c*) *Method of Measuring.*—The average thickness shall be taken as one-half the difference between the mean of the maximum and minimum diameters measured at any one point, and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement made over the conductor plus the thinnest wall, and the diameter of

the conductor. (The first measurement is made by "slicing off" the heavier side of the insulation.)

The measurements may be made with any type of micrometer, reading to at least 0.001 in., which is suitable for measurements of this character.

In the case of multiple-conductor cable, the measurements shall be made on the individual wires before being cabled.

(*d*) *Number of Measurements.*—When the lot of wire to be inspected consists of two coils or reels or less, at least one determination of the thickness shall be made on each coil or reel. When the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of the two coils or reels taken at random. If the lot consists of 20 coils or reels or over, at least one determination of the thickness shall be made on each lot of not less than 10 per cent of the total number of coils or reels.

(*e*) *Rejection.*—If the thickness of any coil or reel is found to be less than the prescribed value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

V. CHEMICAL PROPERTIES AND TESTS.

14. (*a*) *Composition.*—The insulation shall contain exclusively Hevea rubber which has not been previously used, waxy hydrocarbons consisting of ceresine or refined solid paraffin, sufficient sulfur to properly vulcanize, and fillers which are entirely inorganic mineral matter containing no red lead, carbon or bitumen. **Composition of Compound.**

(*b*) *Requirements.*—The vulcanized compound shall conform on analysis to the following requirements expressed as percentages, by weight, of the whole sample:

	MAXIMUM.	MINIMUM.
Rubber	33	30
Waxy hydrocarbons	4	..
Free sulfur	0.7	..

Results shall be taken between the limits given in proportion to the percentage by weight of the rubber found.

The limits allowed for 30-per-cent rubber compound shall be as follows:

	MAXIMUM.	MINIMUM.
Saponifiable acetone extract	1.35	0.55
Unsaponifiable resins	0.45	....
Chloroform extract	0.90	....
Alcoholic potash extract	0.55	....
Total sulfur (see note)	2.10	....
Specific gravity	....	1.75

The limits allowed for 33-per-cent rubber compound shall be as follows:

	Maximum.	Minimum.
Saponifiable acetone extract	1.50	0.60
Unsaponifiable resins	0.50	....
Chloroform extract	1.00	....
Alcoholic potash extract	0.60	....
Total sulfur (see note)	2.30	....
Specific gravity	....	1.67

The acetone solution shall not fluoresce.

The acetone extract (60 cc.) shall not be darker than a light straw color.

Hydrocarbons shall be solid, waxy and not darker than a light brown.

Chloroform extract (60 cc.) shall not be darker than a straw color.

Contamination of the compound, such as by the use of impregnated tapes, will not excuse the manufacturer from conforming to these specifications.

(*c*) The analysis of the compound shall be made in accordance with Sections 15 to 47, inclusive (Joint Rubber Insulation Committee's Procedure) of the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

NOTE.—The limit on total sulfur may be omitted at the option of the purchaser.

(*d*) *Samples.*—Samples for chemical analysis shall be taken before the application of the braid and after the material has passed all physical and electrical tests. The purchaser may take samples in accordance with Section 18. The purchaser shall assure himself that all samples are free from contamination and change due to torch heating. Wherever practicable, samples shall be taken at a distance of at least 3 ft. from the end of the wire.

(*e*) *Retests and Rejections.*—The purchaser may make a chemical analysis on any one of the samples selected as above to determine if the compound meets the requirements of Paragraph (*b*). Failure of any one sample selected shall be sufficient cause to reject the wire which the sample represents, except in the case of failure to meet the requirements of either or both free sulfur and chloroform extract in accordance with Paragraph (*b*). In case of such failure the coil from which the sample was taken shall be rejected and two additional samples taken from the remainder of the order. The remainder of the order shall be accepted if both samples so selected shall meet the requirements for free sulfur or chloroform extract in accordance with Paragraph (*b*). The entire order shall be rejected if either sample fails to meet the requirements.

[1] 1924 Book of A.S.T.M. Standards.

VI. PHYSICAL PROPERTIES AND TESTS.

15. The test specimen shall have the following physical properties: **Physical Properties.**

Tensile strength, minimum, lb. per sq. in.	1000
Set, maximum in 2 in., in.	$\frac{3}{8}$
Elongation, minimum stretch before rupture, in.	2 to 9

16. (*a*) *Tension Test.*—A test specimen having a length of not less than 6 in., marked with bench marks 2 in. apart, shall be placed in the jaws of a testing machine (maximum distance between jaws holding the test specimen, 4 in.) and stretched at the rate of 20 in. per minute (jaw speed) until the specimen breaks. The test specimen shall break between the bench marks and the tensile strength shall be calculated upon the area of the original sample. **Physical Tests.**

(*b*) *Set Test.*—A second test specimen having a length of not less than 6 in., marked with bench marks 2 in. apart, shall be placed in the jaws of a testing machine (maximum distance between jaws holding test specimen, 4 in.) and stretched at a rate of 20 in. per minute until the bench marks are 6 in. apart. After the termination of this stretch, the test specimen shall be released within 5 seconds and the set determined 1 minute after the beginning of release.

(*c*) *Elongation Test.*—After the determination of the set in accordance with Paragraph (*b*), the same specimen shall be placed in the jaws of a testing machine (maximum distance between jaws holding test specimen, 4 in.) and stretched at the rate of 20 in. per minute (jaw speed) until the specimen breaks. The test specimen shall break between the bench marks.

(*d*) *Accuracy of Machine.*—The testing machine shall be accurate within 1 per cent of the breaking load.

(*e*) *Jaws.*—The jaws of the machine shall be of an approved type.

(*f*) *Temperature.*—All physical tests shall be made at a room temperature of from 18 to 29.5° C. (65 to 85° F.) inclusive, and the test specimen shall have been kept at the room temperature not less than 30 minutes prior to the tests.

17. (*a*) *Test Specimen.*—The test specimen may be the entire section of the insulation in the case of small wires, or in the case of a large wire, a segment of a section, cut with a sharp knife held tangentially to the conductor. The test specimen shall be as free as possible from surface incisions and imperfections. **Test Specimen.**

NOTE.—The removal of the rubber insulation can be greatly accelerated and in most cases a test specimen which is an entire section can be obtained free from

surface incisions and imperfections by means of metallic mercury. The mercury should be introduced at one end of the sample between the insulation and the tinned surface of the conductor, and the sample inclined on a support with the end to which the mercury is applied at the top. The separation of the rubber insulation results from the amalgamation of the tin of the conductor with the mercury. The amalgamation is assisted by first immersing and rubbing the tinning on the exposed end of the conductor in the mercury.

(*b*) *Buffing.*—In event of any irregularities on the surface of the test specimen, it shall be made smooth and of uniform thickness within 5 per cent of the original thickness by buffing, except when large strands are used, in which case the rubber sample shall be buffed sufficiently to remove all corrugations.

(*c*) *Limits.*—There shall be no limit to the cross-section of the test specimen, except as restricted by the capacity of the testing machine.

(*d*) *Calculation of Area.*—Calculation of the area of the test specimen shall be made as follows:

1. When the total cross section of the insulation is used, the area shall be calculated as the area of the circle whose diameter is the minimum average outside diameter of the insulation minus the area of the conductor. The area of a stranded conductor shall be figured from its maximum diameter.

2. When a slice cut from the insulation by a knife held tangent to the wire is used, and the slice so cut has the cross-section of a segment of a circle, the area shall be calculated as that of the segment of a circle whose diameter is that of the insulation. The height of the segment is the wall of insulation on the side from which the slice is taken. (The values may be easily obtained from a table giving the areas of segments of a unit circle for the ratio of the height of the segment to the diameter of the circle.

3. When the cross-section of the slice is not a segment of a circle, the area shall be calculated from a direct measurement of the volume or from the specific gravity and the weight of a known length of a uniform cross-section.

4. When a portion of a sector of a circle has to be taken where the conductor is large and the insulation thin, the area shall be calculated as the thickness times the width. (This applies either to a straight test piece or one stamped out with a die, and assumes that corrugations have been removed by buffing.)

5. When a portion of a sector of a circle has to be taken where the conductor is large and the insulation thick, the area shall be calculated as the proportional part of the area of the total cross-section.

18. (*a*) When an order calls for less than 500 ft., physical tests may be waived at the option of the purchaser. **Number of Physical Test Samples.**

(*b*) When an order calls for 500 ft. or more and is made into one or more coils, reels or lengths, samples shall be taken from 10 per cent of the coils, reels or lengths, but in any case at least one sample shall be taken.

19. (*a*) *Retests.*—If a specimen fails to pass in one of the three tests prescribed in Section 15, two more specimens shall be taken from the same sample and the average of the results shall determine whether or not the sample complies with the requirements. **Retests and Rejections, Physical Tests.**

(*b*) *Rejections.*—When ten or more samples are selected in any inspection lot, all coils, reels or lengths shall be rejected if more than 10 per cent of the samples fail. If 10 per cent or less fail, each coil, reel or length may be tested and shall be accepted or rejected upon the results of such individual test. Where the number of samples selected in any inspection lot is less than ten, all coils, reels or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel or length may be tested and shall be accepted or rejected upon the result of individual test.

VII. ELECTRICAL PROPERTIES AND TESTS.

20. (*a*) *Place.*—Electrical tests on the wire and cable shall be made at the place of manufacture. **Electrical Properties.**

(*b*) *Requirements.*—Every coil, reel or length of wire, after vulcanization and before the application of paraffin or any covering other than tape used in vulcanization, shall successfully withstand a high voltage test as specified in Table V and shall have an insulation resistance test not less than that specified in Table VI. These tests shall be made after not less than 12 hours immersion in water, and while still immersed.

TABLE V.—VALUES OF TEST POTENTIALS FOR WORKING PRESSURES UP TO 600 VOLTS. A. C.

(AMERICAN INSTITUTE OF ELECTRICAL ENGINEERS)

SIZE, A.W.G. OR CIR. MILS.	SIZE, SQ. MM.	TEST PRESSURE, KILOVOLTS.
14–8 A.w.g.	2.081 – 8.366	3.0
7–0000 A.w.g.	10.55 – 107.2	3.5
250 000 cir. mils and larger.......	127 and larger	4.0

For Working Pressures over 600 volts, A.C.—One kilovolt per $\frac{1}{64}$ in. of thickness (2.53 kv. per mm.) up to $\frac{10}{64}$ in. (3.96 mm.). Above $\frac{10}{64}$ in. (3.96 mm.), the test pressure shall be 10 kilovolts plus 1.5 kilovolts per $\frac{1}{64}$ in. (3.79 kv. per mm.) additional up to $\frac{30}{64}$ in. (11.89 mm.). Where the insulation thickness is $\frac{16}{64}$ in. (6.34 mm.) or over, this rule shall apply only to conductors over 26,000 cir. mils (13.2 sq. mm.) area.

High Voltage Test. 21. The test voltage shall be applied for 5 minutes. The test shall be made in accordance with the Standardization Rules of the American Institute of Electrical Engineers. (See Appendix.)

Insulation Resistance. 22. (*a*) *Method.*—The insulation resistance test shall be made in accordance with the Standardization Rules of the American Institute of Electrical Engineers and within the range of temperature given in Table VII. (See Appendix.)

(*b*) *Temperature Coefficient.*—The insulation resistance (megohms) shall be reduced to that at 15.55° C. (60° F.) by multiplying by the coefficient in Table VII corresponding to the temperature at which the test is made.

TABLE VI.—INSULATION RESISTANCE, IN MEGOHM-MILES AT 15.55° C. (60° F.).[1]

Size of Conductor.	Thickness of Insulation in sixty-fourths of an inch.																
	2	3	4	5	6	7	8	10	12	14	16	18	20	22	24	26	28
18 A.w.g.	1600	2050	2450	2800	3000	3200	3400	3750	4050	4300	4500	4700	4900	5000	5150	5300	5400
16 "	1400	1800	2150	2400	2650	2850	3050	3400	3650	3950	4150	4350	4500	4650	4800	4900	5050
14 "	..	1530	1850	2150	2350	2550	2650	3050	3350	3550	3800	3950	4100	4300	4400	4550	4650
12 "	..	1350	1600	1850	2050	2250	2400	2750	3000	3250	3400	3600	3750	3900	4050	4150	4300
10 "	..	1150	1350	1600	1800	2000	2150	2400	2650	2900	3100	3250	3400	3550	3700	3800	3900
8 "	..	850	1050	1250	1450	1650	1750	2050	2200	2400	2600	2750	2900	3050	3150	3250	3350
6 "	..	..	850	1050	1200	1350	1450	1750	1950	2100	2300	2450	2550	2700	2800	2900	3000
4 "	..	..	750	850	1000	1150	1250	1450	1650	1850	2000	2150	2250	2400	2500	2600	2700
2 "	..	..	650	750	850	950	1050	1250	1450	1600	1700	1850	1950	2100	2200	2300	2400
1 "	..	..	600	650	750	850	1000	1150	1350	1450	1600	1750	1850	1950	2050	2150	2250
0 "	..	..	..	600	700	800	950	1050	1200	1350	1500	1600	1700	1800	1900	2000	
00 "	..	..	..	550	650	750	850	950	1050	1250	1350	1500	1600	1700	1800		
000 "	..	..	..	500	600	650	750	850	1000	1150	1250	1350	1450	1550			
0 000 "	..	..	..	450	550	600	650	800	950	1050	1150	1250	1350	1450			
	..	..	..														
250 000 cir. mil	..	..	..	400	475	575	625	725	875	1000	1100	1200	1275				
350 000 "	..	..	..	350	425	475	525	675	775	850	950	1050	1125				
500 000 "	..	..	..	300	375	400	475	575	675	750	850	900					
750 000 "	..	..	..	..	..	325	400	475	525	625	700	775					
1 000 000 "	..	..	..	..	..	300	325	400	475	550	625						
1 250 000 "	..	..	..	..	..	..	325	375	425	500	575						
1 500 000 "	..	..	..	..	..	..	300	325	400	475							
1 750 000 "	..	..	..	..	..	..	275	300	375	450							
2 000 000 "	..	..	..	..	..	..	200	275	325	425							

NOTE.—For intermediate sizes, use values of next larger size.

[1] This table is based on the constant 4000 in the following formula:

$R = K \log_{10} \frac{D}{d}$, where R = resistance in megohms, K = constant, D = diameter over insulation, d = diameter over conductor.

Rejections, Electrical Test. 23. Every coil, reel or length shall be rejected if it fails to comply with the electrical requirements herein specified.

(*C*) RUBBER-FILLED CLOTH TAPE.

Material. 24. The tape shall be made from cotton cloth having a weight of not less than one pound per four yards with a width of 36 in. and not less than 56 by 60 picks per in., and shall be thoroughly filled with a rubber compound.

Application. 25. The tape shall be applied helically. The maximum width and minimum overlap shall conform to the values in Table VIII.

TABLE VII.—TEMPERATURE COEFFICIENTS.

Temperature.		Coefficient.	Temperature.		Coefficient.
Deg. Fahr.	Deg. Cent.		Deg. Fahr.	Deg. Cent.	
46	7.8	0.694	61	16.1	1.026
47	8.3	0.709	62	16.7	1.053
48	8.9	0.729	63	17.2	1.081
49	9.4	0.746	64	17.8	1.109
50	10.0	0.769	65	18.3	1.138
51	10.6	0.787	66	18.9	1.169
52	11.1	0.806	67	19.4	1.200
53	11.7	0.833	68	20.0	1.231
54	12.2	0.854	69	20.6	1.264
55	12.8	0.877	70	21.1	1.297
56	13.3	0.900	71	21.7	1.331
57	13.9	0.917	72	22.2	1.366
58	14.4	0.943	73	22.8	1.402
59	15.0	0.970	74	23.3	1.438
60	15.6	1.000	75	23.9	1.477

TABLE VIII.—WIDTH AND OVERLAP FOR RUBBER-FILLED CLOTH TAPE.

Diameter Over Insulation, in.	Width, Maximum, in.	Overlap, Minimum, in.
2.00	5	$\frac{1}{2}$
1.75	$4\frac{1}{2}$	$\frac{1}{2}$
1.50	4	$\frac{1}{2}$
1.25	$3\frac{1}{2}$	$\frac{1}{2}$
1.15	$3\frac{1}{4}$	$\frac{1}{2}$
1.00	3	$\frac{1}{2}$
0.88	$2\frac{3}{4}$	$\frac{3}{8}$
0.75	$2\frac{3}{8}$	$\frac{3}{8}$
0.62	2	$\frac{3}{8}$
0.50	$1\frac{3}{4}$	$\frac{3}{8}$
0.38	$1\frac{1}{2}$	$\frac{1}{4}$
0.31	$1\frac{1}{4}$	$\frac{1}{4}$
0.25	1	$\frac{1}{4}$
0.19	$\frac{7}{8}$	$\frac{1}{8}$
0.16	$\frac{3}{4}$	$\frac{1}{8}$

Intermediate Diameters. 26. For intermediate diameters the requirements shall be those of the next smaller diameter indicated in Table VIII.

Number of Tests. 27. Samples shall be taken in accordance with Section 18 or at the option of the purchaser.

(*D*) BRAID.

Material. 28. The braid shall be closely woven from cotton thread having not less than two plys and shall be completely impregnated with an insulating waterproof compound and finished with a black insulating compound thoroughly slicked down.

Compound. 29. The compound shall be neither injuriously affected by nor have injurious effect upon the braid at a temperature of 90° C. (194° F.).

Construction. 30. (*a*) The braid shall be so constructed that the tangent of the angle between the cotton thread and the side of the wire shall be not

TABLE IX.

Diameter Under Braid, in.	Minimum Closeness Constant (k).	Minimum Thickness of Braid (t), in.
$0-\frac{7}{32}$	6	0.018
$\frac{8}{32}-\frac{14}{32}$	6	0.020
$\frac{15}{32}-\frac{20}{32}$	7	0.020
$\frac{21}{32}-\frac{25}{32}$	8	0.024
$\frac{26}{32}$ and greater	8	0.024

less than 0.70. The values of the closeness constant, k, and the thickness of braid, t, shall be not less than those in Table IX.

(*b*) The values given in Table IX shall be based upon the following formulae:

$$A = \frac{\pi p D}{a}$$

$$t = 2d$$

$$k = \frac{abt}{D}\sqrt{1+A^2}$$

in which

A = tangent of angle between the cotton thread and the side of the wire;

t = thickness of braid in inches;

k = closeness constant;

D = diameter of wire under braid in inches;

a = number of carriers;
b = number of ends per carrier
p = two times the picks per inch = number of carriers per inch;
d = average diameter of single thread of cotton per inch.

(*c*) The various quantities shall be determined as follows:

D, by a suitable micrometer after removal of the braid;

a and b, by examination of the braid;

p, on a 4 in. length of sample after wiping off the outside of the braid with chloroform;

d, by measuring 25 per cent of the total number of threads with a paper micrometer having a foot 0.4 in. in diameter, under a pressure of 50 g. The sample of braid on which the average diameter is determined can be prepared by immersing a 2 in. length of sample in chloroform in a suitable container for 5 minutes, decanting and repeating for 1 minute, and removing and drying it thoroughly by pressing it between cheese cloth.

Number of Tests

31. Samples shall be taken in accordance with Section 18 or at the option of the purchaser.

(*E*) INSPECTION.

Inspection and Test.

32 (*a*) Inspection shall be made prior to shipment and at the place of manufacture.

(*b*) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the wire or cable to permit of arrangement of inspection.

(*c*) The inspector representing the purchaser shall have free entry at all times, while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the wire ordered, except compounding room. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the wire is being furnished in accordance with these specifications.

(*d*) The purchaser at his option may make the various tests on samples in his own laboratory or elsewhere, but such tests shall be made at the expense of the purchaser.

Rehearing.

33. Samples of rejected material shall be preserved for two weeks from date of test report. In case of dissatisfaction with the results of test, the manufacturer may make claim for a rehearing within that time. Sealed duplicate samples may be retained by the manufacturer at his option.

APPENDIX.

EXTRACTS FROM STANDARDIZATION RULES OF THE AMERICAN INSTITUTE OF ELECTRICAL ENGINEERS.

(Edition, 1921.)

Conductivity of Copper.

1. The following shall be taken as normal values for standard annealed copper:

(*a*) At a temperature of 20° C. the resistance of a wire of standard annealed copper 1 m. in length and of a uniform section of 1 sq. mm. is $\frac{1}{58}$ ohm = 0.017241 ohm.

(*b*) At a temperature of 20° C. the density of standard annealed copper is 8.89 g. per cc.

(*c*) At a temperature of 20° C. the "constant mass" temperature coefficient of resistance of standard annealed copper, measured between two potential points rigidly fixed to the wire, is 0.00393 = 1/254.45 . . . per degree Centigrade.

(*d*) As a consequence, it follows from (*a*) and (*b*) that at a temperature of 20° C. the resistance of a wire of standard annealed copper of uniform section, 1 m. in length and weighing 1 g. is $\frac{1}{58} \times 8.89$ = 0.15328 ohm.

High Voltage Test.

(*a*) *Frequency.*

2. The frequency of the test voltage shall not exceed 100 cycles per second, and should approximate as closely as possible a sine wave. The source of energy should be of ample capacity.

(*b*) *Rate of Increase of Voltage.*

3. The initially-applied voltage shall not be greater than the working voltage and the rate of increase shall not be over 100 per cent in 100 seconds.

(*c*) *Multiple Conductor Cables.*

4. If a multiple conductor cable is designed for the same operating voltage between conductors and sheath, or water, as between conductors, each conductor shall be tested against the other conductors

connected together and to the sheath, or water. If the cable is designed for an operating voltage between conductors and ground different from that between conductors, the test between conductors and the sheath, or water, shall be made separately and shall be based on the normal operating voltage between conductors and sheath, or water, as prescribed for single-conductor cables.

INSULATION RESISTANCE TEST.

(*a*) *Electrification.*

5. The apparent insulation resistance should be measured after the dielectric-strength test, measuring the leakage current after a one-minute electrification, with a continuous e.m.f. of from 100 to 500 volts, the conductor being maintained negative to sheath, or water.

(*b*) *Multiple Conductor Cables.*

6. The insulation resistance of each conductor of a multiple-conductor cable shall be the insulation resistance measured from each conductor to all the other conductors in multiple with the sheath or water.

TENTATIVE SPECIFICATIONS

FOR

STEAM HOSE[1]

Serial Designation: D 54 - 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1921; Revised, 1924.

Material Covered.

1. These specifications cover wrapped fabric steam hose suitable for steam connections when the steam pressure does not exceed 125 lb. per sq. in.

I. MANUFACTURE

Construction.

2. The hose shall consist of:

(*a*) An inner rubber tube;

(*b*) Cotton duck reinforcement;

(*c*) An outer rubber cover;

(*d*) A protective covering of wire armor when specified.

Rubber Tube.

3. The inner rubber tube shall be smooth, uniform in quality and thickness, and free from injurious defects.

Table I.—Number of Plies

Inside Diameter of Hose, in.	Number of Plies
$\frac{3}{8}$, $\frac{1}{2}$, $\frac{3}{4}$	4
1, $1\frac{1}{4}$, $1\frac{1}{2}$, $1\frac{5}{8}$	5
$1\frac{3}{4}$	6
2	8

Cotton Reinforcement.

4. (*a*) The reinforcement shall consist of plies of cotton duck cut on the bias and applied evenly and firmly over the tube with not less than a $\frac{1}{2}$-in. lap, not sewed. The number of plies shall be as specified in Table I. The plies of duck shall be well frictioned on both sides with a rubber compound which shall firmly join the plies to the rubber tube and cover and to each other.

(*b*) The cotton duck shall be evenly woven from high-grade cotton and shall be free from mechanical defects.

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[1] These Tentative Specifications are in effect a revision of the Standard Specifications for Steam Hose. The Standard Specifications, which were last published under the Serial Designation: D 54 - 20, have accordingly been withdrawn.

Criticisms of these Tentative Specifications are solicited and should be directed to Mr. E. H. Grafton, Secretary of Committee D-11 on Rubber Products, Quaker City Rubber Co., Wissinoming, Philadelphia, Pa.

(c) The reinforcements shall be sufficiently strong to enable the hose to successfully withstand the prescribed hydrostatic pressure test, yet at the same time be soft and pliable.

Rubber Cover. 5. The rubber cover shall be uniform in quality and thickness, and free from injurious defects.

Wire Armor. 6. The wire for wire-wound hose shall be galvanized steel, half-round or elliptical, $\frac{7}{32}$ in. in diameter, wound tightly around the outside of the hose with a pitch of 1 in.

II. PHYSICAL PROPERTIES AND TESTS

Standard Methods. 7. The tests necessary to determine the physical properties which are prescribed in the following sections shall be made in accordance with the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1]

Test Samples. 8. (a) Two samples, each 24 in. in length, shall be taken from each lot of 500 ft. or less of each size of long-length hose and from each lot of 200 pieces or less of car-heating steam hose. One of these samples shall be used for hydrostatic tests and the other for the physical and steam tests.

(b) A portion of sufficient length shall be cut from one of the samples and subjected to tension, elongation and friction tests. The remaining portion of the sample shall be used for the steam tests.

Tests of Tube and Cover. 9. (a) In the case of hose 1 in. or less in diameter, the test specimens shall be cut longitudinally from the sample of hose; in the case of hose over 1 in. in diameter the test specimens shall be cut transversely.

(b) The tensile strength and elongation of the tube and cover shall not be less than the following values:

	Before Steam Test	After Steam Test
Tensile strength of tube.......	750 lb. per sq. in.	550 lb. per sq. in.
Tensile strength of cover......	600 lb. per sq. in.	450 lb. per sq. in.
Elongation at rupture.........	2 in. to 6 in.	2 in to 4 in.

Friction Tests. 10. The adhesion between plies, between the cover and the plies, and between the tube and the plies shall be such that the weights specified below will not cause a separation at a rate greater than 1 in. per minute:

	Weight, lb. Before Steam Test	Weight, lb. After Steam Test
Between plies of duck.......................	18	15
Between duck and tube.....................	15	12
Between duck and cover....................	15	12

[1] 1924 Book of A.S.T.M. Standards.

Maximum Deterioration after Steam Test.

11. After the steam test, the tensile strength and elongation of the tube and cover shall not be less than the values specified in Section 9 (*b*). Furthermore, for the tube these values shall not be more than 50 per cent less than the values obtained before steaming and for the cover not more than 60 per cent less than the values obtained before steaming.

Steam Test.

12. (*a*) The rack steam test shall be used. The test length described in Section 8 (*b*) shall be connected to a rack and dry saturated steam at an average pressure of 75 lb. per sq. in. ± 3 lb. shall be allowed to pass through the hose continuously for two periods of seven hours each. Between the first and second periods and between the second period and the time of testing, the hose shall be permitted to rest not less than 12 nor more than 18 hours.

(*b*) The hose upon examination immediately after its removal from the rack shall disclose no blistering of the tube or loosening of the tube from the fabric nor shall the inside diameter be more than 15 per cent greater than the original diameter.

TABLE II.—HYDROSTATIC TEST PRESSURES

INSIDE DIAMETER OF HOSE, IN.	TEST PRESSURES, LB. PER SQ. IN.
$\frac{3}{8}$, $\frac{1}{2}$	800
$\frac{3}{4}$	750
1	750
$1\frac{1}{4}$	750
$1\frac{1}{2}$	650
$1\frac{5}{8}$	650
$1\frac{3}{4}$	650
2	550

Hydrostatic Tests.

13. The 24-in. section selected as described in Section 8 (*a*) shall be subjected to a hydrostatic test and shall not burst at a lower pressure than that specified in Table II for hose of its size.

III. SIZES AND DIMENSIONS

Sizes and Dimensions.

14. (*a*) The hose shall be in accordance with the size and length specified by the purchaser.

(*b*) The thickness of the cover shall not be less than $\frac{1}{16}$ in. The thickness of the tube shall not be less than $\frac{1}{8}$ in.

(*c*) The diameters of the hose shall be in accordance with Table III. The inside diameter shall not vary more than $\pm\frac{1}{32}$ in. and the outside diameter not more than $\pm\frac{1}{16}$ in. from the values given.

IV. MARKING

Marking.

15. (*a*) Each 50-ft. length of hose shall have inlaid in rubber in three places, namely, midway between the ends and within 10 ft. of each end, a brand showing the name of the manufacturer, the month and year of manufacture, the trade name of the hose, and the legend

"A.S.T.M. Specifications." In addition, each brand shall bear a serial number for each lot of 500 ft. or less of each size of long-length hose and for each lot of 200 pieces or less of car-heating steam hose, the serial number to begin with 1 on the first of each year and continue consecutively until the end of the year. Lengths shorter than 50 ft. shall have only one brand similar to the above at a point near the center of the length. The letters and figures shall be at least $\frac{1}{4}$ in. high, shall stand $\frac{1}{32}$ in. in relief, and shall be clear and distinct.

(*b*) Serial numbers of rejected hose shall not be applied to any other hose during the same calendar year.

V. INSPECTION AND REJECTION

Inspection. 16. (*a*) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the hose to permit of arrangement for inspection.

TABLE III.—DIAMETERS

Nominal Inside Diameter, in.	Nominal Outside Diameter, in.
$\frac{3}{8}$	1
$\frac{1}{2}$	$1\frac{1}{8}$
$\frac{3}{4}$	$1\frac{1}{2}$
1	$1\frac{13}{16}$
$1\frac{1}{4}$	$2\frac{1}{16}$
$1\frac{1}{2}$	$2\frac{5}{16}$
$1\frac{5}{8}$	$2\frac{7}{16}$
$1\frac{3}{4}$	$2\frac{5}{8}$
2	$3\frac{1}{16}$

(*b*) The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the hose is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*c*) The purchaser may make the tests to govern the acceptance or rejection of the hose in his own laboratory or elsewhere. But such tests shall be made within 60 days after receipt of the shipment and at the expense of the purchaser.

Rejection. 17. (*a*) Each length of hose which fails to meet any of the requirements of these specifications shall be rejected.

(*b*) In the case of failure to pass any one of the tests specified in Sections 9 to 13, inclusive, the entire lot of hose represented by the sample, or samples, subjected to these tests, shall be rejected.

Rehearing. 18. Samples of rejected hose shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
RUBBER INSULATING TAPE.[1]

Serial Designation: D 119 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1921; REVISED, 1922.

Material Covered. 1. These specifications cover rubber insulating tape to be used for insulating joints in electric wires and cables.

I. MANUFACTURE.

Compound 2. The tape shall be a rubber compound which shall be well, evenly and smoothly calendered, cut to uniform width and tightly wound in rolls with a glazed cloth, parchment paper or a linen separator interposed between adjacent layers.

Separator. 3. The separator shall be attached to and cover the outer side of the tape. When unwound from the original roll, it shall show no undue tendency to stick to the rubber.

II. CHEMICAL PROPERTIES AND TESTS.

Composition. 4. The tape shall be a rubber compound containing not less than 30 per cent of the best quality Hevea rubber, not more than one per cent of free sulfur and not more than 4 per cent of waxy hydrocarbons.

The remainder shall consist only of suitable dry inorganic mineral fillers. The compound shall contain no reclaimed rubber, substitutes or organic matter other than herein above specified. All percentages shall be based on the weight of the original compound.

Chemical Analysis. 5. If an analysis of the compound is made, it shall be carried out in accordance with Sections 15 to 47, inclusive (Joint Rubber Insulation Committee's Procedure), of the Standard Methods of Testing Rubber

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. E. H. Grafton, Secretary of Committee D-11 on Rubber Products, Quaker City Rubber Co., Wissinoming, Philadelphia, Pa.

Products (Serial Sesignation: D 15) of the American Society for Testing Materials[1], with the following exceptions:

(*a*) Omit "chloroform extract" from the diagram in Fig. 1 and from the tabulation in Section 45.

(*b*) In place of Section 20 substitute the following: "Remove the rubber compound from the separator and cut into as thin strips as possible."

(*c*) Omit Sections 25 and 33.

(*d*) In Section 34, substitute "acetone" for "chloroform" in the first and second lines.

(*e*) In Section 38, omit "from the conductor" in the third and fourth lines.

(*f*) In Section 46, omit "and chloroform" and substitute "extract" for "extracts" in the first sentence. Omit the second sentence.

(*g*) In Section 47, omit "chloroform extract."

III. PHYSICAL PROPERTIES AND TESTS.

Tensile Strength.

6. (*a*) The tensile strength of the tape shall be not less than 300 lb. per sq. in.

(*b*) The tests shall be made with a power driven, pendulum-type tension testing machine; the initial distance between the jaws shall be 3 in. and the rate of separation of the jaws shall be 20 in. per minute. The width of the test specimen shall be 0.5 in.

(*c*) Three specimens of each sample free from visible flaws shall be tested. The average result shall be taken as the tensile strength.

(*d*) The temperature of the room shall be not lower than 65° F. (18° C.) nor higher than 90° F. (32° C.) and the samples shall have been kept within these temperature limits for at least 30 minutes previous to the time of testing.

Dielectric Strength.

7. Two brass-ball sphere electrodes 2 cm. in diameter shall be brought so close together that the sample of tape can be just moved between them. The tape shall withstand without puncturing the application of a potential of 10,000 volts (r.m.s. value) for a period of five minutes applied to the sphere electrodes at a frequency not exceeding 65 cycles per second.

Fusion.

8. The tape when wrapped to a thickness of $\frac{1}{4}$ in. and heated to a temperature of 150° F. (65.5° C.) for 20 minutes shall fuse into a homogeneous mass.

Test Samples.

9. One roll for each 250 rolls shall be taken at random for test. At least 2 ft. of the outer layers shall be removed and discarded before taking specimens for test.

[1] 1924 Book of A.S.T.M. Standards.

IV. STANDARD WEIGHTS, DIMENSIONS AND VARIATIONS.

Thickness, Weight and Yardage.

10. The tape shall conform to the following requirements:

Width, in.	Thickness, in.	Nominal Weight per Roll, lb.	Minimum Net Weight per 100 Rolls, lb.[a]	Minimum Length per Pound, yd.[b]
$\frac{1}{2}$	0.030	$\frac{1}{2}$	50	$26\frac{1}{4}$
$\frac{3}{4}$	0.030	$\frac{1}{2}$	50	$17\frac{1}{2}$
1	0.030	1	100	13

[a] Exclusive of core, wrapping and box.
[b] Exclusive of core, wrapping, box and separator.

Permissible Variations.

11. The width shall not vary from the specified value by more than $\frac{1}{32}$ in. The thickness shall not vary from the specified value by more than 0.003 in.

Measurement of Thickness.

12. The thickness shall be measured with a micrometer graduated to 0.001 in. having a circular foot, 0.24 to 0.26 in. in diameter, and with a pressure on the specimen of not less than 8 oz. or more than 10 oz. Four measurements shall be made at random in a length of not less than 3 ft. and no measurement shall be outside the limits prescribed in Sections 10 and 11.

V. PACKING AND MARKING.

Packing.

13. Each roll shall be wrapped in paraffined paper or metal foil and enclosed in a suitable box. The wrapping shall be secure and shall thoroughly protect the contents.

Marking.

14. Each box shall be marked with the name of the manufacturer or trade mark and the nominal width and weight of the tape.

VI. INSPECTION AND REJECTION.

Time of Inspection.

15. The tape shall be tested and inspected within four weeks of the date of delivery.

Retest and Rejection.

16. If the tape fails in any one test of those prescribed in these specifications, two additional specimens shall be taken and submitted to that test. If either of these two additional specimens fails, the lot of tape represented by that sample roll shall be rejected.

TENTATIVE SPECIFICATIONS

FOR

TEXTILE TESTING MACHINES[1]

Serial Designation: D 76 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1920; REVISED, 1922, 1925.

Type of Machine.

1. Textile testing machines shall be of the inclination balance or pendulum type.

Angle of Pendulum.

2. The maximum angle of swing of the pendulum in textile testing machines shall be 45 deg. from the vertical.

Pendulum Drum.

3. The minimum diameter of drum for transferring the pull on the specimen to the swinging pendulum shall be 2 in.

Testing Machine Capacity.

4. In selecting the proper capacity of a textile testing machine for a given sample of fabric or yarn, the maximum capacity of the machine shall not exceed that at which the pendulum reaches a swing of 45 deg. from the vertical. The minimum capacity of the machine when used for a given sample of fabric or yarn shall not be less than 20 per cent of the above maximum capacity.

Type of Jaw.

5. (*a*) *Fabric Jaws.*—The clamps of textile testing machines for use upon fabrics shall consist of flat metallic jaws pressing directly against the specimen. One gripping surface shall be hinged or swiveled and the other shall be rigidly connected to the frame of the jaw. The pressure between the jaws shall be secured by any suitable mechanical device so constructed as to grip the fabric firmly before the testing load is applied and to prevent visible slippage during the progress of the test.

(*b*) *Skein Jaws.*—The drums of testing machines for yarn skeins shall consist of cylindrical spools not less than 1 in. in diameter and not less than 1 in. in width, so supported that at least one shall turn freely upon its axis.

(*c*) *Individual Strand Jaws.*—The jaws or clamps for tests upon individual strands of yarn shall be of the cylindrical or drum type, so

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, United States Rubber Co., 122 Adams St., Newark, N. J.

arranged that the strands of yarn shall pass around not less than 180 deg. circumference before being clamped or fixed in the jaw. The length of the specimen shall be considered from center to center of drums. The minimum diameter of the cylinder or drum shall be $\frac{1}{2}$ in.

Jaw Dimensions. 6. The width of jaw in a direction perpendicular to the specimen shall in no case be less than 1 in. The depth of jaw in a direction length-wise of the specimen shall in no case be less than 1 in.

Backlash. 7. The dial pointer of textile testing machines shall be so arranged as to be easily adjustable to a zero reading for any weight of jaw or other fixture in the testing machine. The dial pointer shall be so counter-weighted as to prevent undue fluctuations in its position due to backlash, whatever the dial reading may be.

Machine Speed. 8. Textile testing machines shall be power driven or operated in such a manner as to produce a uniform and accurate movement of 12 in. per minute for the pulling jaw.

Calibration. 9. In calibrating textile testing machines, dead weights of accurate amounts shall be used, but these weights shall be applied at a speed of 12 in. per minute, corresponding to the standard jaw speed. The machine shall otherwise be arranged in an entirely similar manner to that used in testing fabric.

TENTATIVE SPECIFICATIONS
FOR
TOLERANCES AND TEST METHODS FOR COTTON YARNS, SINGLE AND PLIED[1]

Serial Designation: D 180 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision

ISSUED, 1923; REVISED, 1924, 1925.

Scope. 1. These specifications cover the tolerances and methods of testing for cotton yarn, both single and plied.

TOLERANCES

I. STRENGTH

Strength of Yarn. 2. The average tensile strength of each case, bale, chain ball or beam warp of yarn, either single or plied, as found by test shall be not less than the specified strength.

II. SIZE OR YARN NUMBER

Size of Yarn, Carded and Combed. 3. The average size of each case, bale, chain ball or beam warp of yarn, in the singles, either carded or combed, as found by test shall not vary more than 3 per cent above or below the specified size.

Example.—No. 36 carded yarn specified; tolerance would allow 34.92 to 37.08.

III. TWIST AND DIRECTION OF TWIST

Definition. 4. *Direction of Twist.*—In the case of yarn, the yarn has right-hand twist if, when it is held vertically, the spirals or twists are seen to incline upward in a right-hand direction, and has left-hand twist when the spirals or twists are seen to incline upward in a left-hand direction.

NOTE.—The definition as here given does not agree with the definition of "direction of twist" of cotton sewing thread as appearing in the Tentative Specification of Tolerances and Test Methods for Cotton Sewing Threads (D 204 – 24 T) which definition defines "direction of twist" in such a way that right-hand twist in the case of sewing thread corresponds to left-hand twist in the case of yarn, as defined herein. An attempt is being made to harmonize these two conceptions of "direction of twist."

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, U. S. Rubber Co., 122 Adams St., Newark, N. J.

Twist of Plied Yarns.

5. The average twist of each case, bale, chain ball or beam warp of plied yarns as found by test shall not vary more than 5 per cent above or below that specified.

Example.—Twist specified is 20 twists per inch; tolerance 19 to 21.

METHODS OF TESTING

IV. STRENGTH

Two test methods are given, the skein test for single yarn and the single strand test for single and plied yarns. A Preferred and Alternative Method for each test is given. The Alternative Method can be used where routine testing is done on a large scale. The Preferred Method should always be used in case of dispute.

Strength, Single Yarn,—Skein Preferred.

6. *Skein Test (Preferred Method).*—A standard skein (120 yd.) shall be broken after conditioning of tubes or bobbins selected for test for 12 hours or of skeins for at least 3 hours in an atmosphere of 65 per cent relative humidity, 70° F. (21° C.). An automatic power yarn tester of inclination balance type, the maximum capacity of which shall be determined in accordance with a table of machine specifications, shall be used. The drums of the testing machine shall consist of cylindrical spools not less than one inch in diameter and not less than one inch in width, so supported that at least one shall turn freely upon its axis. The speed of the pulling jaw shall be 12 in. per minute. Any yarn reel having a $1\frac{1}{2}$-yd. perimeter may be used in preparing the skeins. For filling-wound yarns or yarns on cones, where the yarn is drawn from the top, a speed of 100 to 300 r.p.m. of reel shall be used. For warp-wound yarns or yarn on parallel tubes where the yarn is drawn from the side, a speed of 20 to 30 r.p.m. of reel shall be used. On reels that have only one pigtail guide, the tension shall be applied by making one full wrap of the yarn around the guide. On reels using two or more guides, the yarn shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension. Judgment must be used in regard to the amount of tension required on yarns having little or a large amount of twist. Three tests from each of four bobbins from every case of yarn shall be made.

Strength, Single Yarn, Single Strand Preferred.

7. *Strength Yarn on Beams (Skein Test for Yarn on Beams).*—Yarn received on beams shall be given the skein test and skeins shall be prepared in the following manner: Place the beam containing the yarn to be tested on two bearings sufficiently high from the floor so that the beam can be turned easily. Directly in front of the beam and at least 15 ft. from the beam, a small table shall be placed, the top of which will be approximately the same height as the top of the beam of yarn. On this table a standard yarn reel shall be placed directly in front of the beam. A small crank arm shall be attached

to one end of the beam shaft. Four ends of yarn, taken from the beam, shall be attached to the yarn reel in the regular manner, such as is done when testing yarn from bobbins. One operative shall turn the beam slowly in the proper direction so that the yarn will unwind, and the second operative shall turn the reel fast enough to take up the yarn as it comes off the beam. This operation shall be continued until 120 yd. or the desired length for testing has been taken from the beam. The four skeins shall then be taken from the reel and tested in the regular manner. If it should be necessary to test more ends, the same method of testing shall be followed.

NOTE.—In using these beams after these ends have been taken off, a set of spools containing the same kind of yarn as is on the beam can be placed in behind the beam on a small creel, to fill out the number of ends that have been used for testing. When the ends come up on the beams that have been used for testing, the auxiliary spools in back of the beams can be broken out.

8. *Single Strand Test* (*Preferred Method*).—Single strands shall be broken after conditioning the tubes or bobbins for 12 hours in an atmosphere of 65 per cent relative humidity, 70° F. (21° C.). A single strand tester of proper capacity with the jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of 4 breaks from each of 10 bobbins shall be the average strength.

Strength, Plied Yarns, Single Strand, Preferred.

9. *Plied Yarns* (*Preferred Method*).—Plied yarns, except standard tire cord, shall be subjected to the single strand break after conditioning for 12 hours on spools or tubes selected for test, in an atmosphere of 65 per cent relative humidity and 70° F. (21° C.). Standard tire cord shall be tested under dry conditions in accordance with the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1] A single strand tester of proper capacity with the jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of 4 breaks from each of 10 spools or tubes shall be reported as the average strength.

Strength for All Yarns, Alternative Method.

10. *Skein and Single Strand* (*Alternate Method*).—Skeins or single strands of yarn either single or plied, prepared in accordance with Sections 6, 7, 8 and 9, shall be broken under natural humidity conditions at time of test. The results thus obtained shall be reduced to a common basis of standard moisture regain equal to 7 per cent of the bone-dry weight.

Moisture Regain Determination.

11. To determine moisture regain present in samples, the several skeins shall be weighed collectively, immediately after testing, under natural moisture conditions which obtain at the time of test. The skeins shall then be placed in the basket of an oven at a temperature

[1] 1924 Book of A.S.T.M. Standards.

of 105 to 110° C. (221 to 230° F.) and dried to constant weight. The moisture regain is then computed as the percentage of the dry weight.

Correction to Standard Regain.

12. (*a*) The following formula shall then be applied, based on the assumption that the standard moisture regain of cotton yarns is 7 per cent of the dry weight, that the actual percentage regain is between the limits of 3 and 7 per cent of the dry weight and that for 1 per cent of moisture regain there is an increase of 6 per cent in the tensile strength of the yarn.

$$\text{Tensile strength corrected to standard moisture regain} = \frac{\text{(Tensile strength from machine reading)} \times 142}{100 + (6 \times \text{actual percentage regain.})}$$

Example.—With a skein of yarn broken under natural conditions showing a tensile strength of 120 lb. as read from the machine dial, and which by weighing before and after drying the skein was found to contain a moisture regain equal to 5 per cent of the bone-dry weight, the tensile strength corrected to a common basis of 7 per cent moisture regain would be:

$$\text{Corrected tensile strength} = \frac{120 \times 142}{100 + (6 \times 5)} = 131 \text{ lb.}$$

(*b*) Moisture regain tests shall be made periodically during the hours of testing as the natural humidity conditions are found to vary.

Strength Correction to Size.

13. The average tensile strength shall be corrected to the specified size as determined in accordance with Sections 14, 15 and 16, by the following formula:

$$\text{Corrected tensile strength} = \text{Actual average strength} \times \frac{\text{Actual average size}}{\text{Specified size}}$$

V. SIZE OR YARN NUMBER

Size of Single Yarns, Preferred Method.

14. The size of all standard skeins used in the skein strength test described in Section 6 shall be determined immediately after being broken. In case the single strand test is made, the standard skein shall be prepared for the size determination at the time of the break and the size determined immediately. The size of 60-yd. bundles prepared from beams as described in Section 7 shall be determined as soon as possible after conditioning and one test from each beam shall be reported as the average size. The balance to be used in this test shall be accurate to 0.25 per cent of the standard size of the yarn. When the balance does not indicate the size directly, the yarn number or size may be calculated from the formula:

$$\text{Yarn number or size} = \frac{\text{Length in yards of single yarn}}{\text{Weight in grains}} \times \frac{7000 \text{ (grains in 1 lb.)}}{840 \text{ (yards of No. 1 cotton yarn per pound)}}$$

15. In determining the size of plied yarns, the skein shall be prepared in accordance with Table I, and the size shall be determined after conditioning of tubes or spools selected for test for 12 hours or of skeins for at least 3 hours, in an atmosphere of 65 per cent relative humidity, and 70° F. (21° C.). Any yarn reel having a $1\frac{1}{2}$-yd. perimeter may be used in preparing the skeins. For filling-wound yarns or yarn on cones, a speed of 100 to 300 r.p.m. of reel shall be used. For warp-wound yarns or yarn on parallel tubes, a speed of 20 to 30 r.p.m. of reel shall be used. On reels that have only one pigtail guide, the tension shall be applied by making one full wrap of the yarn around the guide. On reels using two or more guides, the yarn shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension. **Size of Plied Yarns, Preferred Method.**

TABLE I.

Equivalent Singles Size	Yards for Size	Conversion Formula	Number of Tests Per Case of Yarn
20's and above	60	$\frac{\text{Size}}{2} = \text{Ply size}$	3 from each of 4 spools or tubes
3's to 20's	24	$\frac{\text{Size}}{5} = \text{Ply size}$	3 from each of 4 spools or tubes
Below 3's	12	$\frac{\text{Size}}{10} = \text{Ply size}$	3 from each of 4 spools or tubes

16. All yarns used in the alternative method of testing for strength (Section 10) shall be sized under natural humidity conditions at the time of test. Plied yarns shall be prepared in skeins in accordance with Table I. The moisture regain shall then be determined as specified in Section 11, and results corrected to a common basis of standard moisture regain equal to 7 per cent of the bone-dry weight by means of the formula: **Size of All Yarns, Alternate Method.**

$$\text{Size corrected to standard moisture regain} = \frac{\text{Size} \times (100 + \text{actual percentage regain})}{107}$$

The average of these tests shall be the average size of case, bale, ball chain or beam warp of yarn.

VI. TWIST

17. No precision method for determining the twist of single yarns has been developed. **Twist of Single Yarns.**

18. The ply twist in yarns of 2 or more ply shall be determined on any standard twist counter with jaws set 10 in. apart. The strands shall be clamped in jaws under a definite tension by attaching weights. **Twist of Plied Yarns.**

The tension to be used shall be determined from the formula:

$$\text{Tension, in grams} = \frac{156 \text{ (Constant)}}{\text{Equivalent singles size}}$$

Example.—The weight for 23/11 would be as follows:

$$\frac{156}{2.09} = 76 \text{ g.}$$

The constant of 156 represents a tension which should be placed on yarn or cord to hold it sufficiently taut and still not remove any stretch.

Number of Tests. 19. Three twist tests on each of 4 packages of yarn from each case shall be made, and the average of these 12 tests shall be the average of the case.

TENTATIVE SPECIFICATIONS

FOR

TOLERANCES AND TEST METHODS FOR COTTON SEWING THREADS[1]

Serial Designation: D 204 - 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

TOLERANCES

I. STRENGTH

Strength. 1. The average tensile strength of each case of sewing thread as found by test shall be not less than the specified strength.

II. YARDAGE

Yards per Pound. 2. When thread is sold by weight instead of on a guaranteed yardage unit, the yards per pound shall be indicated on each tube, spool or cone.

Variation. 3. The actual number of yards per pound or unit of sale, as found by test, shall not be more than 4 per cent less than the number of yards per pound or unit of sale specified.

III. TWIST

Definition. 4. *Direction of Twist.*—In the case of sewing thread, the thread as right-hand twist if, when it is held vertically, the spirals or twists are seen to incline upward in a left-hand direction, and has left-hand twist if the spirals or twists are seen to incline upward in a right-hand direction.

NOTE.—The definition as here given does not agree with the definition of "direction of twist" of yarn as appearing in the Standard Specifications for Tolerances and Test Methods for Electrical Cotton Yarns (D 203 - 25) and in the Tentative Specifications for Tolerances and Test Methods for Cotton Yarns, Single and Plied (D 180 - 25 T) which definitions define "direction of twist" in such a way that left-hand twist in the case of yarn corresponds to righ-hand twist in the case of sewing thread, as defined herein. An attempt is being made to harmonize these two conceptions of "direction of twist."

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, U. S. Rubber Co., 122 Adams St , Newark, N. J.

Balance.

5. The finished sewing thread shall be balanced in twist as nearly as possible and in no case shall a yard of thread when drawn from the side of a spool or package twist upon itself more than six times when brought together to form a loop.

IV. RUNNING QUALITIES

Running Qualities.

6. In addition to meeting the foregoing requirements, sewing thread shall be subjected to a sewing machine test under actual factory conditions. In the event of any thread apparently failing to operate properly, it shall be judged by comparison on any given machine under identical conditions, with thread from a lot known to have previously operated satisfactorily.

METHODS OF TESTING

V. STRENGTH

Strength.

7. All threads shall be tested by the single strand test method. A single strand tester of proper capacity with jaws set 10 in. between grips and having a speed of pulling jaw of 12 in. per minute shall be used. The average of ten breaks from each of five spools or packages shall be the average strength. Any test during which the thread breaks at the jaw due to pinching or cutting shall not be included in the average.

VI. YARDAGE

Yards per Pound.

8. In determining the yards per pound of sewing thread, the skeins shall be prepared in accordance with Table I. Any yarn reel having a $1\frac{1}{2}$-yd. perimeter may be used in preparing the skeins. On reels that have only one pigtail guide, tension shall be applied by making one full wrap of the thread around the guide. On reels using two or more guides, the thread shall pass straight through the guides onto the reel, the angles of the guides supplying the necessary tension.

Table I.

Equivalent Single Size	Yards for Test	Number of Tests per Case of Thread
Finer than 10's................	120	3 from each of 4 spools
10's and coarser...............	60	3 from each of 4 spools

The skeins shall then be weighed and the average weight of the 12 skeins shall be converted to yards per pound from the formula:

$$\text{Yards per lb.} = \frac{7000 \times (\text{number of yards in skein})}{\text{Average weight in grains of skein}}$$

VII. LENGTH OF PACKAGE

Length of Package.

9. (*a*) (*Preferred Method*).—The length of package of sewing thread shall be determined by multiplying the number of yards per

pound, obtained as outlined in Section 8, by the net weight in pounds of the thread in package to be tested.

(b) (*Alternate Method*).—The length of package of sewing thread shall be determined by measuring the entire length over a reel having a $1\frac{1}{2}$-yd. perimeter, taking three full turns around the reel and delivering thread to a take-up drum. The yardage shall be noted on reel dial.

(c) (*Alternate Method*).—The length of package of sewing thread shall be determined by measuring the thread in the form of skeins prepared on a reel having a $1\frac{1}{2}$-yd. perimeter. In no case shall the yardage of these skeins exceed that specified in Table II. The total yardage of skeins thus prepared from a package constitutes its length.

TABLE II.

EQUIVALENT SINGLE SIZE	LENGTH OF SKEIN, YD.
Finer than 12's	240
6's to 12's, inclusive	120
Coarser than 6's	60

VIII. TWIST AND BALANCE

10. No precision method for determining the twist of single yarns has been developed. **Twist in Singles.**

11. Ply twist shall be determined on any standard twist counter with the jaws set 10 in. apart. The strands shall be clamped in jaws under a definite tension obtained by attaching weights. The tension to be used shall be determined from the formula: **Twist in Ply.**

$$\text{Tension, in grams} = \frac{156 \text{ (Constant)}}{\text{Equivalent singles size}}$$

Example.—The weight for 24/4 would be as follows:

$$\frac{156}{6} = 26 \text{ g.}$$

The constant of 156 represents a tension which should be placed on yarn or thread to hold it sufficiently taut and still not remove any stretch.

12. The "balance" of a sewing thread shall be determined by removing one yard of thread from a spool or package, holding it at each end and bringing these ends together, allowing the thread a chance to loop. **Balance.**

TENTATIVE SPECIFICATIONS

FOR

TOLERANCES FOR NUMBERED COTTON DUCK[1]

Serial Designation: D 230 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Tolerances. 1. Tolerances shall be the limit within which a textile must come in its specified characteristics in order that it shall constitute a good delivery on contract. They may be classified as the allowable limits of the quantitative characteristics of the fabric as defined in the specifications. The following tolerances are based upon the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[2]

I. WIDTH

Width. 2. The average width determined by measurement shall be as specified with the following tolerances:

Width, in.	Tolerance, in. Under	Tolerance, in. Over
Up to and including 36	$\frac{1}{4}$	$\frac{1}{4}$
37 to 60, inclusive	$\frac{1}{4}$	$\frac{3}{8}$
61 to 80, inclusive	$\frac{3}{8}$	$\frac{5}{8}$
81 to 120, inclusive	$\frac{3}{8}$	$\frac{3}{4}$

II. WEIGHT

Weight. 3. The weight of the fabric determined by test shall be not more than 2.5 per cent over or under the specified weight.

III. THREADS PER INCH

Threads per Inch. 4. (*a*) The average count of warp ends per inch determined by test shall be not more than $1\frac{1}{2}$ ends over or under the specified count for fabrics counting not over 40 ends per inch and not more than 2 ends over or under for fabrics counting over 40 ends per inch.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, United States Rubber Co., 122 Adams St., Newark, N. J.

[2] 1924 Book of A.S.T.M. Standards.

(b) The average count of filling picks per inch determined by test shall be not more than 1 pick over or under the specified count for fabrics counting not over 25 picks per inch, and not more than $1\frac{1}{2}$ picks over or under for fabrics counting from $25\frac{1}{2}$ to 32 picks per inch, and not more than 2 picks over or under for fabrics counting over 32 picks per inch.

IV. STRENGTH

5. The average tensile strength of the warp and the average tensile strength of the filling shall be not less than the average specified tensile strength of each. **Strength.**

TENTATIVE SPECIFICATIONS

FOR

TOLERANCES AND TEST METHODS FOR KNIT GOODS[1]

Serial Designation: D 231 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. These specifications are intended to be applicable to the examination of those classes of knit goods which later enter into a manufacturing process. Where a material requires special treatment, specific methods will be described as they are developed for that material and such special tests will have precedence over these general specifications.

TOLERANCES

I. WIDTH

Width. 2. The width as determined by test shall not be more than 3.5 per cent above or 3.5 per cent below the specified width.

II. WEIGHT

Weight. 3. The weight of the fabric as determined by test shall be not more than 5 per cent over or under the specified weight.

III. COUNT

Count. 4. The average count of wales per inch and courses per inch as determined by test shall be not more than 5 per cent over or under the specified count.

IV. TENSILE STRENGTH

Tensile Strength. 5. The average tensile strength of the fabric as determined by test shall be not less than the specified tensile strength.

METHODS OF TESTING

V. MOISTURE REGAIN

Moisture Regain. 6. (*a*) The moisture present shall be determined as follows: Portions of the samples which are taken for the determination of weight (as described in Section 10 (*a*)) weighing from 3 to 5 g. shall

[1] Criticism of these Tentative Specifications are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, U. S. Rubber Co., 122 Adams St., Newark, N. J.

be placed in any suitable receptacle and the weight determined before and after drying in a ventilated oven at 105° C. The drying shall be continued until there is no change in weight, due care being taken to desiccate the receptacle and contents until cool, before weighing.

(*b*) The loss in weight shall be considered moisture regain and the percentage estimated on the sample in bone-dry condition.

(*c*) Two determinations should be sufficient for ordinary purposes. Additional determinations may be required if desired.

VI. DETERMINATION OF GREASE

Determination of Grease.

7. (*a*) The amount of grease present shall be determined as follows: A representative sample which weighs about 5 g. taken from the samples prepared for strength tests shall be dried to constant weight in a ventilated oven at 105° C. It shall then be extracted with carbon tetrachloride in an apparatus as shown in Fig. 1, Section 8, of the Standard Methods of Testing Rubber Products (Serial Designation: D 15) of the American Society for Testing Materials.[1] The extraction shall be continued for 8 hours and the extract dried to constant weight in a weighed flask at 105° C.

(*b*) The extract shall be considered grease and the percentage shall be reported on the dried sample.

(*c*) Two determinations of grease are usually sufficient. Additional determinations may be required if desired.

VII. PERCENTAGE OF COTTON AND WOOL

Percentage of Cotton and Wool.

8. (*a*) The amount of cotton and wool present shall be determined by the so-called "caustic-potash" method. This method shall be carried out as follows: The sample which has been dried for moisture determination shall be boiled thoroughly in 1-per-cent hydrochloric acid, but not long enough to disintegrate the fibers. This shall be repeated until no residue is found upon evaporating the solution on a watch glass. This removes sizing, dyestuff, mineral loading soluble in hydrochloric acid, etc. The residue shall be extracted successively with alcohol and carbon tetrachloride until all soluble material is removed. It shall be dried at 105° C. and weighed. This weight represents combined cotton and wool.

The dried residue shall be treated with a solution of 5-per-cent caustic potash and boiled gently for 15 minutes. It shall be poured into 500 cc. of cold water; permitted to stand until the fibers have settled and the supernatant liquid decanted. It shall be filtered through a Gooch crucible, washed with hot water and finally with

[1] 1924 Book of A.S.T.M Standards.

10 cc. of alcohol. It shall then be dried and weighed. The loss in weight represents wool; the residue represents cotton, except that a correction shall be made for 3.5 per cent soluble cotton.

Example:

Preliminary Analysis		
Sample weight	5.000 g.	
Moisture	0.4370 g.	8.74 per cent
Sizing, grease, etc	0.1640 g.	3.28 "
Fibers (by difference)	4.3990 g.	87.98 "
Analysis of Bone Dry Fibers		
Wool (loss to caustic potash)	2.3200 g.	52.74 "
Cotton (residue)	2.0790 g.	47.26 "
Cotton (corrected) (2.0790÷0.965)	2.1544 g.	48.97 "
Wool (corrected)	2.2446 g.	51.03 "
Air Dry Analysis		
Wool plus normal regain	2.5588 g.	50.99 "
Cotton plus normal regain	2.2945 g.	45.73 "
Sizing, grease, etc	0.1640 g.	3.27 "

Assumptions	
Normal regain for wollen goods	14 per cent on dry weight
Normal regain for cotton goods	6.5 " " " "
Cotton soluble in caustic potash	3.5 " " " "

(*b*) Two determinations of percentage of cotton and percentage of wool are usually sufficient. Additional determinations may be required if necessary.

VIII. WIDTH

Width. 9. (*a*) The width of the roll or piece (either split or tubular) shall be determined at five different places uniformly distributed along the full length of the roll or piece. The goods shall be drawn across a table without tension when the width is measured.

(*b*) The average of the five measurements shall be the width.

IX. WEIGHT

Weight. 10. (*a*) Five samples which have been died out in staggered fashion across the roll or piece shall be weighed collectively. The die shall be square and 2 in. on a side. The material shall not be under tension as it is sampled.

(*b*) The samples shall be weighed under prevailing atmospheric conditions except in the settlement of disputes. In such cases, tests shall be made upon samples which have normal moisture content, obtained by exposure for at least 4 hours to an atmosphere of 65 per cent relative humidity at a temperature of 70° F. (21° C.); or upon

agreement, the samples shall be weighed in the dry condition as described in Section 2 of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39) of the American Society for Testing Materials.[1]

(*c*) The weight shall be reported as weight per square yard as calculated from the weight found.

X. THICKNESS

11. The thickness shall be measured by an automatic micrometer as described in Section 11 of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39). **Thickness.**

XI. WALES AND COURSES

12. The number of wales and courses per inch shall be determined by counting a space of not less than 2 in. in at least five different places in the roll or piece. The material shall be laid out on a table without tension while the wales and courses are being counted. **Wales and Courses.**

XII. TENSILE STRENGTH

13. (*a*) The tensile strength shall be determined on an approved type of inclination balance breaking machine. The maximum capacity of the machine shall be such that the position of the pendulum shall not be greater than 45 deg. from the vertical when the break occurs. The minimum capacity shall not be less than 20 per cent of the above maximum capacity. **Tensile Strength.**

(*b*) The samples shall be tested under prevailing atmospheric conditions except in the settlement of disputes. In such cases, tests shall be made upon samples which have normal moisture content, obtained by exposure for at least 4 hours to an atmosphere of 65 per cent relative humidity at a temperature of 70° F. (21° C.); or upon agreement, the tests shall be carried out upon samples which have been bone-dried as described in Section 2 of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39).

(*c*) The 1 by 1 by 1-in. grab method shall be used as described in Section 19 (*d*) of the Standard Methods of Testing Cotton Fabrics (Serial Designation: D 39), with the single exception that the distance between jaws shall be changed from 3 in. to 1 in.

(*d*) At least 20 test pieces 4 in. in width by 6 in. in length shall be cut, ten in the direction of the wales and ten in the direction of the courses.

(*e*) The tensile strength in each direction shall be the average of the ten tests.

[1] 1924 Book of A.S.T.M. Standards.

TENTATIVE METHODS OF TESTING COTTON FIBERS.[1]

Serial Designation: D 152 – 22 T.

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

ISSUED, 1922.

Group Strength Test Method.

1. The strength shall be the strength of a group of fibers expressed in terms of a weight equal to a unit weight of 20s yarn.

FIG. 1.—Brushing to Remove Short Fibers.

I. PREPARATION OF SAMPLE.

Preparation of Sample.

2. (*a*) The cotton shall be pulled down in the customary manner of a cotton classer which produces fibers that are essentially parallel.

(*b*) The fibers shorter than $\frac{7}{8}$ in. shall be removed by means of a small brush. See Fig. 1.

(*c*) The group or bundle of fibers shall be cut to equal $\frac{7}{8}$ in. to assure all fibers being $\frac{7}{8}$ in. long. See Fig. 2.

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, U. S. Rubber Co., 122 Adams St., Newark, N. J.

(*d*) The bundles shall be weighed, each bundle to weigh approximately 0.004 g.

(*e*) The ends of each group shall be cemented with collodion to prevent the fibers from slipping. See Fig. 3.

Five bundles shall be used.

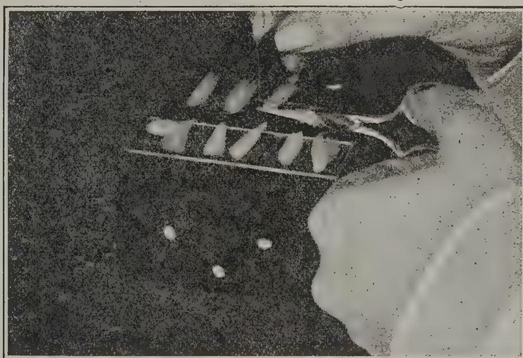

FIG. 2.—Cutting Fibers to Equal Length.

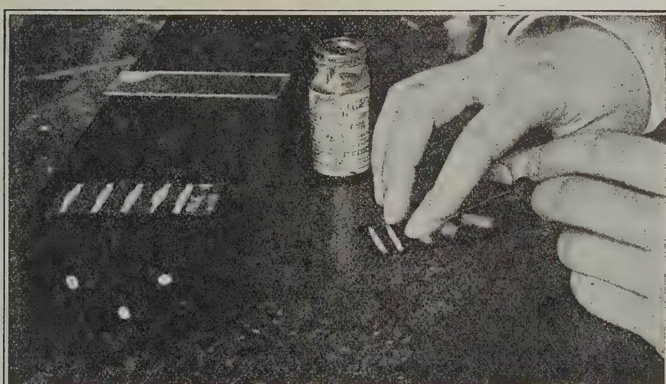

FIG. 3.—Cementing Ends of Fiber Groups.

II. PROCEDURE.

3. The samples shall be exposed to a relative humidity of 70 per cent for two hours before weighing and before testing for strength. **Moisture Condition.**

4. The conventional inclination balance testing machine of 10-lb. capacity shall be used with the clamp moving at the rate of 12 in. per minute. **Machine.**

The distance between the clamps shall be $\frac{1}{2}$ in.

The clamps shall be flat, of the metal or rubber insert type, $\frac{1}{2}$ in. in width and $\frac{1}{2}$ in. in depth.

Result. 5. The result shall be expressed in terms of an equivalent weight of 20s yarn by the formula:

$$\text{Strength in ounces of cotton equal to unit of 20s yarn} = \frac{\text{Sum of Strengths in pounds} \times 16 \times 453.6 \times 7}{\text{Sum of Weights in grams} \times 840 \times 20 \times 36 \times 8}$$

TENTATIVE METHODS OF TESTING GREASE WOOL AND ALLIED FIBERS FOR SCOURED CONTENT[1]

Serial Designation: D 232 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Scope. 1. These methods of test are intended to be applicable for testing grease wool and allied fibers (such as mohair, alpaca, camel hair, cashmere, vicuna) which contain grease, sand, dirt and vegetable matter, for the scoured content.

Form of Material. 2. The scoured content may be determined on the following forms of loose fiber in the grease:

(*a*) Wool (etc.) in the fleece;
(*b*) Necks, pieces, bellies;
(*c*) Pulled wool;
(*d*) Colonial scoured.

I. WOOL (ETC.) IN THE FLEECE

3. The tests to determine the scoured content of grease wool in the fleece shall be made on not less than 500 lb. but need not be on more than 1000 lb.

Weight. 4. (*a*) The wool shall be weighed in the original bags or bales at the time and place of making the test. This shall be considered the gross weight.

(*b*) The weight of the coverings and bindings and the string used for tying the fleeces, known as the tare, shall be determined.

(*c*) The difference between the gross weight and the tare shall be the "normal net weight" of the grease wool.

Sorting. 5. To retain the full commercial value of the wool after scouring it shall be sorted previous to scouring into an agreed number of main and off sorts.

Tags, Fribs, Clips. 6. The tags, fribs, clips and paint clips shall not be scoured, but shall be weighed in the grease and reported separately as a percentage of the normal net weight of the grease wool.

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, United States Rubber Co., 122 Adams St., Newark, N. J.

Scouring or Washing. 7. The main sorts and off sorts excepting tags, fribs, clips and paint clips, shall be scoured separately in a train of at least 4 bowls, or in 3 bowls and a rinse box. The first 3 bowls shall contain the detergents and the last bowl or the rinse box shall contain clean running water.

Drying. 8. The sorts shall be thoroughly dried in a heated mechanical dryer or on a heated table dryer and shall be bagged and weighed at once.

Hot Weight. 9. The total net weight of all the lots from the dryer, known as the "hot weight" of the scoured wool shall be determined.

Hot Shrink. 10. The difference between the normal net weight of the grease wool and the hot net weight of the scoured wool shall be determined as the "hot shrink" of the grease wool, when expressed as a percentage of the normal net weight of the grease wool by the following formula:

$$\text{Percentage Hot Shrink} = \frac{\begin{array}{c}(\text{Normal Net Weight of Grease Wool}-\\ \text{Hot Net Weight of Scoured Wool}) \times 100\end{array}}{\text{Normal Net Weight of Grease Wool}}$$

Moisture in Hot Net Weight. 11. To determine the moisture in the hot net weight, three samples of not less than 1 lb. each shall be taken from the bags or bag representing about the beginning, the middle and the end of the run, immediately after the hot weight has been determined, and immediately placed in dry air-tight containers, the weights of which have been previously determined, and carefully weighed therein (check weight being made on containers); the net weight calculated, and the samples dried to constant weight in a ventilated drying oven maintained at a temperature of 230 to 240° F. (110 to 116° C.) as shown by two consecutive weighings not less than ten minutes apart without removing from the oven.

Bone Dry Weight. When two such weighings do not show a further loss of more than 0.1 per cent of the previous weighing the material is considered to be bone dry.

Actual Regain in Hot Weight. 12. The difference between the average of the original (initial) weights of the samples, and the average of the bone dry weights of the same, shall be considered to be the moisture loss.

(*a*) When computed as a percentage of the initial weight it is the moisture content percentage.

(*b*) When computed as a percentage of bone dry weight it is the moisture regain percentage.

Standard Condition or Conditioned Weight. 13. The standard condition of scoured wool shall be understood to be the condition in which it contains 12 per cent of its dry weight of moisture.

14. This 12 per cent shall be the standard regain of scoured wool. **Standard Regain.**

15. The conditioned weight of the scoured wool shall be calculated from the following formula: **Formula for Conditioned Weight.**

$$\text{Conditioned Weight} = \frac{\text{Hot Net Weight} \times 112}{100 + \text{Actual Regain}}$$

16. The true yield of the test lot shall be calculated from the following formula and shall be the actual yield of the entire lot. **Conditioned Yield.**

$$\text{Yield, per cent} = \frac{\text{Conditioned Weight of Scoured Wool} \times 100}{\text{Normal Net Weight of Grease Wool}}$$

17. If the loss in scouring is to be expressed as shrinkage, it shall be calculated from the following formula and shall be the actual shrinkage of the entire lot. **Conditioned Shrinkage.**

$$\text{Shrinkage, per cent} = \frac{(\text{Normal Net Weight of Grease Wool} - \text{Conditioned Weight of Scoured Wool}) \times 100}{\text{Normal Net Weight of Grease Wool}}$$

18. Wool shall be considered thoroughly scoured when tests by the Soxhlet Extraction Apparatus do not show a loss of more than one per cent of the weight of the conditioned scoured wool before testing. **Thoroughly Scoured Wool.**

19. Grease and solubles not to exceed a fixed percentage may be left in scoured wool by agreement, and tests to determine this percentage shall be made by the Soxhlet Extraction Apparatus. **Scoured Wool Containing Grease.**

II. NECKS, PIECES AND BELLIES

20. The tests to determine the scoured content of necks, pieces and bellies shall be made on not less than 500 lb. but need not be on more than 1000 lb. wool in the grease.

21. (*a*) The wool shall be weighed in the original bags or bales at the time and place of making the tests. This shall be considered the gross weight. **Weight.**

(*b*) The weight of the coverings and bindings of the bales, known as the tare, shall be determined.

(*c*) The difference between the gross weight and the tare shall be the "normal net weight" of the grease wool. **Normal Net Weight.**

22. The test for scoured content shall be made as described for wool in the fleece in accordance with Sections 5 to 19, inclusive. **Scoured Content.**

III. PULLED WOOL

23. The tests to determine the scoured content of pulled wool shall be made on not less than 500 lb. but need not be on more than 1000 lb. of wool in the grease.

Weight. 24. (*a*) The wool shall be weighed in the original bags or bales at the time and place of making the tests. This shall be considered the gross weight.

(*b*) The weight of the coverings and bindings, known as the tare, shall be determined.

(*c*) The difference between the gross weight and the tare shall be the "normal net weight" of the grease wool.

Scouring or Washing. 25. The wool shall be scoured in a train of at least 4 bowls or in 3 bowls and a rinse box. The first 3 bowls shall contain the detergents and the last bowl or rinse box shall contain clean running water.

Drying. 26. The wool shall be thoroughly dried in a heated mechanical dryer or on a heated table dryer and shall be bagged and weighed at once.

Hot Weight. 27. The net weight of the wool from the dryer, known as the "hot weight" of the scoured wool shall be determined.

Scoured Content. 28. The remainder of the test for determining the scoured content shall be made as described for wool in the fleece in accordance with Sections 10 to 19, inclusive.

IV. COLONIAL SCOURED

29. The test to determine the scoured content of colonial scoured wool shall be made as described for pulled wool in accordance with Sections 23 to 28, inclusive.

TENTATIVE DEFINITIONS OF TERMS RELATING TO TEXTILE MATERIALS[1]

Serial Designation: D 123 – 22 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and until its adoption as Standard it is subject to revision.

Issued, 1921; Revised, 1922.

(*A*) *Mechanical Fabric*

1. The term "Mechanical Fabric" shall be understood to mean fabric manufactured for use as an intermediate product in the making of some mechanically constructed article.

2. Mechanical fabrics shall be referred to by the following descriptive nomenclature:

(*a*) A word or phrase generally descriptive of the appearance of the material, or the use to which it is put.

(*b*) Weight in ounces per square yard. The words "oz. sq. yd." are to be written after the weight. To avoid confusion in regard to weight of material, it is suggested that immediately succeeding the square yard weight, the weight of the material on whatever basis may have been used previous to the adoption of these standards be inserted in brackets. Table I gives the conversion values of linear yard weight to square yard weight.

(*c*) Width in inches.

(*d*) Count, giving first the ends, and second the picks per inch.

Example: Hose Duck, 9 oz. sq. yd. [40 in. 10 oz.], 48 in., 28 by 18.

3. The following is a partial list of mechanical fabrics in general use:

Tire Builder Fabric.—A square woven fabric having usually 11-ply yarn in both warp and filling.

Tire Cord Fabric.—A fabric consisting of hawser cord yarn in the warp, with single yarn filling at intervals to keep warp threads together.

Hose Duck.—A soft, plain-weave fabric of plied yarns not finer than No. 8, made in weights from 10 to 24 oz. to the 40-in. width. When made of finer yarn than No. 8, it is classed as Special Hose Duck.

Rubber Belt Duck.—A soft, plain-weave fabric of plied yarns not finer than No. 8, weight ranging from 22 to 36 oz. to 42-in. width. When made of finer yarn than No. 8, it is classed as Special Belt Duck.

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[1] Criticisms of these Tentative Definitions are solicited and should be directed to Mr. K. B. Cook, Secretary of Committee D-13 on Textile Materials, U. S. Rubber Co., 122 Adams St., Newark, N. J.

TABLE I.—CONVERSION TABLE OF LINEAR YARD WEIGHT TO SQUARE YARD WEIGHT OF FABRIC.

Weight, oz.	Width, in.																													
		31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
8......	9.60	9.29	9.00	8.73	8.47	8.23	8.00	7.78	7.58	7.38	7.20	7.02	6.86	6.70	6.55	6.40	6.26	6.13	6.00	5.88	5.76	5.65	5.54	5.43	5.33	5.24	5.14	5.05	4.97	4.88	4.80
9......	10.80	10.45	10.13	9.82	9.53	9.26	9.00	8.76	8.53	8.31	8.10	7.90	7.71	7.53	7.36	7.20	7.04	6.89	6.75	6.61	6.48	6.35	6.23	6.11	6.00	5.89	5.79	5.68	5.59	5.49	5.40
10......	12.00	11.61	11.25	10.91	10.59	10.29	10.00	9.73	9.47	9.23	9.00	8.78	8.57	8.37	8.18	8.00	7.83	7.66	7.50	7.35	7.20	7.06	6.92	6.79	6.67	6.55	6.43	6.32	6.21	6.10	6.00
11......	13.20	12.77	12.38	12.00	11.65	11.31	11.00	10.70	10.42	10.15	9.90	9.66	9.43	9.21	9.00	8.80	8.61	8.43	8.25	8.08	7.92	7.76	7.62	7.47	7.33	7.20	7.07	6.95	6.83	6.71	6.60
12......	14.40	13.94	13.50	13.09	12.71	12.34	12.00	11.68	11.37	11.08	10.80	10.54	10.29	10.05	9.82	9.60	9.39	9.19	9.00	8.82	8.64	8.47	8.31	8.15	8.00	7.85	7.71	7.58	7.45	7.32	7.20
13......	15.60	15.10	14.63	14.18	13.76	13.37	13.00	12.65	12.32	12.00	11.70	11.41	11.14	10.88	10.64	10.40	10.17	9.96	9.75	9.55	9.36	9.18	9.00	8.83	8.67	8.51	8.36	8.21	8.07	7.93	7.80
14......	16.80	16.26	15.75	15.27	14.82	14.40	14.00	13.62	13.26	12.92	12.60	12.29	12.00	11.72	11.45	11.20	10.96	10.72	10.50	10.29	10.08	9.88	9.69	9.51	9.33	9.16	9.00	8.84	8.69	8.54	8.40
15......	18.00	17.42	16.88	16.36	15.88	15.43	15.00	14.60	14.21	13.85	13.50	13.17	12.86	12.56	12.27	12.00	11.74	11.49	11.25	11.02	10.80	10.59	10.38	10.19	10.00	9.82	9.64	9.47	9.31	9.15	9.00
16......	19.20	18.58	18.00	17.45	16.94	16.46	16.00	15.57	15.16	14.77	14.40	14.05	13.71	13.40	13.09	12.80	12.52	12.26	12.00	11.76	11.52	11.29	11.08	10.87	10.67	10.47	10.29	10.11	9.93	9.76	9.60
17......	20.40	19.74	19.13	18.56	18.00	17.49	17.00	16.54	16.11	15.69	15.30	14.93	14.57	14.23	13.91	13.60	13.30	13.02	12.75	12.49	12.24	12.00	11.77	11.55	11.33	11.13	10.93	10.74	10.55	10.37	10.20
18......	21.60	20.90	20.25	19.64	19.06	18.51	18.00	17.51	17.05	16.62	16.20	15.80	15.43	15.07	14.73	14.40	14.09	13.79	13.50	13.22	12.96	12.71	12.46	12.23	12.00	11.78	11.57	11 37	11.17	10.98	10.80
19......	22.80	22.06	21.38	20.73	20.12	19.54	19.00	18.49	18.10	17.54	17.10	16.68	16.29	15.91	15.55	15.20	14.87	14.55	14.25	13.96	13.68	13.41	13.15	12.91	12.67	12.44	12.21	12.00	11.79	11.59	11.40
20......	24.00	23.23	22.50	21.82	21.18	20.57	20.00	19.46	18.95	18.46	18.00	17.56	17.14	16.74	16.36	16.00	15.65	15.32	15.00	14.69	14.40	14.12	13.85	13.58	13.33	13.09	12.86	12.63	12.41	12.20	12.00
21......	25.20	24.39	23.63	22.91	22.24	21.60	21.00	20.43	19.89	19.38	18.90	18.44	18.00	17.58	17.18	16.80	16.43	16.09	15.75	15.43	15.12	14.82	14.54	14.26	14.00	13.75	13.50	13.26	13.03	12.81	12.60
22......	26.40	25.55	24.75	24.00	23.29	22.63	22.00	21.41	20.84	20.31	19.80	19.32	18.86	18.42	18.00	17.60	17.22	16.85	16.50	16.16	15.84	15.53	15.23	14.94	14.67	14.40	14.14	13.89	13.66	13.42	13.20
23......	27.60	26.71	25.88	25.09	24.35	23.66	23.00	22.38	21.79	22.23	20.70	20.20	19.71	19.26	18.82	18.40	18.00	17.62	17.25	16.89	16.56	16.24	15 92	15.62	15.33	15.05	14.79	14.53	14.28	14.03	13.80
24......	28.80	27.87	27.00	26.18	25.41	24.69	24.00	23.35	22.74	22.15	21.60	21.07	20.57	20.09	19.64	19.20	18.78	18.38	18.00	17.63	17.28	16.94	16.62	16.30	16.00	15.71	15.43	15.16	14.90	14.64	14.40
25......	30.00	29.03	28.13	27.27	26.47	25.71	25.00	24.33	23.68	23.08	22.50	21.95	21.43	20.93	20.45	20.00	19.57	19.15	18.75	18.37	18.00	17.65	17.31	16.98	16.67	16.36	16.07	15.79	15.52	15.25	15.00
26......	31.20	30.19	29.25	28.36	27.53	26.74	26.00	25.30	24.63	24.00	23.40	22.83	22.29	21.77	21.27	20.80	20.35	19.91	19.50	19.10	18.72	18.35	18.00	17.66	17.33	17.02	16.71	16.42	16.14	15.86	15.60
27......	32.40	31.35	30.38	29.45	28.59	27.77	27.00	26.27	25.58	24.92	24.30	23.71	23.14	22.60	22.09	21.60	21.13	20.68	20.25	19.84	19.44	19.06	18.69	18.34	18.00	17.67	17.36	17.05	16.76	16.47	16.20
28......	33.60	32.52	31.50	30.55	29.65	28.80	28.00	27.25	26.53	25.85	25.20	24.59	24.00	23.44	22.91	22.40	21.91	21.45	21.00	20.57	20.16	19.76	19.38	19.02	18.67	18.33	18.00	17.68	17.38	17.08	16.80
29......	34.80	33.68	32.63	31.64	30.71	29.83	29.00	28.22	27.47	26.77	26.10	25.46	24.86	24.28	23.73	23.20	22.70	22.21	21.75	21.31	20.88	20.47	20.08	19.70	19.33	18.98	18.64	18.32	18.00	17.69	17.40
30......	36.00	34.84	33.75	32.73	31.76	30.85	30.00	29.19	28.42	27.69	27.00	26.34	25.71	25.12	24.55	24.00	23.48	22.98	22.50	22.04	21.60	21.18	20.77	20.37	20.00	19.64	19.29	18.95	18.62	18.31	18.00
31......	37.20	36.00	34.88	33.82	32.82	31.89	31.00	30.16	29.37	28.62	27.90	27.22	26.57	25.95	25.36	24.80	24.26	23.74	23.25	22.78	22.32	21.88	21.46	21.06	20.67	20.29	19.93	19.58	19.24	18 92	18.60
32......	38.40	37.16	36.00	34.91	33.88	32.91	32.00	31.14	30.32	29.54	28.80	28.10	27 43	26.79	26.18	25.60	25 04	24.51	24.00	23.51	23.04	22.59	22.15	21.74	21.33	20.95	20.57	20.21	19.86	19.53	19.20
33......	39.60	38.32	37.13	36.00	34.94	33.94	33.00	32.11	31.26	30.46	29.70	28.98	28.29	27.63	27.00	26.40	25.83	25.28	24.75	24.24	23.76	23.29	22.85	22.42	22.00	21.60	21.21	20.84	20.48	20.14	19.80
34......	40.80	39.48	38 25	37.09	36.00	34.97	34.00	33.08	32.21	31.38	30.60	29.85	29.14	28.47	27.82	27.20	26.61	26.04	25 50	24.98	24.48	24.00	23.54	23.09	22.67	22.25	21.86	21.47	21.10	20.75	20.40
35......	42.00	40.65	39.38	38.18	37.06	36.00	35.00	34.06	33.16	32.31	31.50	30 73	30.00	29.30	28.64	28.00	27.39	26.81	26.25	25.71	25.20	24.71	24.23	23.77	23.33	22.91	22.50	22.11	21.72	21.35	21.00
36......	43.20	41.81	40.50	39.27	38.12	37.03	36.00	35.03	34.11	33.23	32.40	31.61	30.86	30 14	29.45	28.80	28.17	27.57	27.00	26.45	25.92	25.41	24.92	24.45	24.00	23.56	23.14	22.74	22.34	21.97	21.60

Balata Belt Duck.—A closely woven fabric made of hard twisted plied yarn usually made in wide widths, the weight being based on the square yard.

Oil Belt Duck.—A closely woven fabric of plied yarns not over No. 8, made in a variety of widths, usually a 32-oz. fabric, the weight being based on the square yard.

Numbered Duck.—A plain woven fabric of plied yarns ranging from coarse to fine numbers, the weight being based on 22-in. width.

Army Duck.—A closely woven fabric of plied yarns, usually ranging between 7 to 12 oz., the weight being based on $27\frac{1}{2}$-in. width.

Single Filled or Flat Duck.—A fabric made of single yarn, plain weave, the weight being based on 30-in. width.

Enameling Duck.—A plain woven fabric with laid warp and plied yarn filling, weight being based on $46\frac{1}{2}$-in. width.

(*B*) *Cotton Fabrics Other than Mechanical Fabrics.*

4. *Twill.*—A weave which produces pronounced lines running diagonally across the fabric, some of which are known as follows:

(*a*) *Drills.*—A commercial term for a fabric with warp face twill usually made with three harnesses and single yarns.

(*b*) *Alberts.*—A commercial term for a fabric of twill weave with filling face usually made with four or five harnesses.

(*c*) *Jeans.*—A commercial term for a fabric with warp face twill like a drill but with higher count and finer yarns.

(*d*) *Serge.*—A commercial term for a fabric of twill weave made with four harnesses 2 by 2.

(*e*) *Clay.*—A commercial term for a fabric of twill weave similar to a serge, but made with six harnesses 3 by 3.

(*C*) *Miscellaneous.*

5. *Crimp.*—In the case of fabric, the difference in distance between any two points of the yarn as it lies in the fabric, and the same two points after the yarn has been removed from the fabric and straightened, expressed as a percentage of the distance between the two points as the yarn lies in the fabric.

NOTE.—"Crimp" is not to be confused with "take-up" which is the difference in distance between any two points on the yarn as it lies in the fabric and the same two points after the yarn has been removed from the fabric and straightened, expressed as a percentage of the distance between the two points after the yarn has been straightened. The threads for the "take-up" test shall be straightened with the same machine that is used for the "crimp" test, but for this test it shall be equipped with an extra scale on the dial, in which case the scale shall be plainly marked "crimp" and "take-up."

6. *Off-Square.*—In the case of fabric, the percentage of warp crimp minus the percentage of filling crimp.

TENTATIVE SPECIFICATIONS
FOR
SIEVES FOR TESTING PURPOSES[1]

Serial Designation: E 11 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Sieves for precision testing have a variety of uses, including both dry screening and wet screening of material. The primary object is to separate materials according to the size of particle. Since particles are three dimensional and the openings of screens are areas having two dimensions only, the method of separation by means of screens is at best an approximation. In many instances, however, it is the best practical means at hand, and will give useful and reliable results if care is taken in securing representative samples, in the preparation of the sample for testing, in the details of procedure and in the method of reporting results. In the Appendix are indicated those points which must be considered in specifying sieving tests for any individual material.

An essential in each method, however, is a series of sieves of known characteristics. The important consideration in sieves is the size of opening and with the size of opening known it is possible to translate the results obtained in one series of sieves into terms of another series. The importance of the size of opening in the sieve series covered by these specifications is emphasized by designating the sieves in terms of opening in microns. This new scheme of designation should accordingly cause no confusion, giving as it does a direct indication of the size of opening. As such, it is not open to the objections to the various arbitrary number designations heretofore in use and it is also free from the objection to the designation by means of mesh in that it is independent of the size of wire.

With the size of opening known, the method of recording results is simplified. It is recommended that in reporting sieve analyses in graphic form the sizes of sieve opening be arranged as ordinates with the largest size at the top and that the percentages be plotted as abscissas.

Scope. 1. These specifications cover sieves for use in the usual cases of precision testing for the fineness of materials (classification of materials according to size). The sieves have been designated according to the size of openings in terms of microns, the corresponding United States Standard Sieve Series designation in arbitrary numbers being given for the convenience of those familiar with the latter scheme of designation.

[1] Criticisms of these Tentative Specifications are solicited and should be directed to Mr. F. G. Breyer, Chairman of Sub-Committee, of Committee E-1, on Classification of Material According to Size, New Jersey Zinc Co., Palmerton, Pa.

I. PHYSICAL REQUIREMENTS

Cloth and Frame.

2. Wire cloth for standard sieves shall be woven (not twilled, except that the cloth of 44, 53 and 62-micron sieves may be twilled until further notice) from brass, bronze, or other suitable wire and mounted on the frames without distortion. To prevent the material being sieved from catching in the joint between the cloth and the frame, the joint shall be smoothly filled with solder, or so made that the material will not catch. The sieve frames should be circular, about 20 cm. (8 in.) in diameter and about 5 cm. (2 in.) or 2.5 cm. (1 in.) between the top of the frame and the cloth.

Table I.—Requirements for Sieve Openings and Wire Diameters with Permissible Variations.

Designation	Corresponding U. S. Standard Sieve Series Number	Sieve Opening		Wire Diameter		Tolerance in Average Opening, per cent	Tolerance on Wire Diameter, per cent		Tolerance in Maximum Opening, per cent
		mm.	in.	mm.	in.		Under	Over	
	2	3	4	5	6	7	8	9	10
4760 micron......	4	4.76	0.187	1.27	0.050	±3	15	30	10
4000 "	5	4.00	0.157	1.12	0.044	±3	15	30	10
3360 "	6	3.36	0.132	1.02	0.040	±3	15	30	10
2830 "	7	2.83	0.111	0.92	0.036	±3	15	30	10
2380 "	8	2.38	0.0937	0.84	0.0331	±3	15	30	10
2000 "	10	2.00	0.0787	0.76	0.0299	±3	15	30	10
1680 "	12	1.68	0.0661	0.69	0.0272	±3	15	30	10
1410 "	14	1.41	0.0555	0.61	0.0240	±3	15	30	10
1190 "	16	1.19	0.0469	0.54	0.0213	±3	15	30	10
1000 "	18	1.00	0.0394	0.48	0.0189	±3	15	30	10
840 "	20	0.84	0.0331	0.42	0.0165	±5	15	30	25
710 "	25	0.71	0.0280	0.37	0.0146	±5	15	30	25
590 "	30	0.59	0.0232	0.33	0.0130	±5	15	30	25
500 "	35	0.50	0.0197	0.29	0.0114	±5	15	30	25
420 "	40	0.42	0.0165	0.25	0.0098	±5	15	30	25
350 "	45	0.35	0.0138	0.22	0.0087	±5	15	30	25
297 "	50	0.297	0.0117	0.188	0.0074	±6	15	35	40
250 "	60	0.250	0.0098	0.162	0.0064	±6	15	35	40
210 "	70	0.210	0.0083	0.140	0.0055	±6	15	35	40
177 "	80	0.177	0.0070	0.119	0.0047	±6	15	35	40
149 "	100	0.149	0.0059	0.102	0.0040	±6	15	35	40
125 "	120	0.125	0.0049	0.086	0.0034	±6	15	35	40
105 "	140	0.105	0.0041	0.074	0.0029	±8	15	35	60
88 "	170	0.088	0.0035	0.063	0.0025	±8	15	35	60
74 "	200	0.074	0.0029	0.053	0.0021	±8	15	35	60
62 "	230	0.062	0.0024	0.046	0.0018	±8	15	35	90
53 "	270	0.053	0.0021	0.041	0.0016	±8	15	35	90
44 "	325	0.044	0.0017	0.036	0.0014	±8	15	35	90

Note.—Screens having larger openings than those included in the table may be specified, but in specifying screens of larger openings the actual size of the opening (in millimeters or in inches) shall be stated. A tolerance of ±3 per cent on average openings and of 10 per cent in maximum openings shall be permitted.

Note.—The size specified above is for the regular standard testing sieve, but for special purposes other sizes may be used, as for example, the 3-in. sieve which is designed especially for use on laboratory balances as in the testing of pigments.

Permissible Variations.

3. The average opening between the adjacent warp and the adjacent shoot wires, taken separately, shall be that given in column

3 (or 4) of Table I, within the "Tolerance in Average Opening" given in column 7. The average diameter of the warp and of the shoot wires, taken separately, of the cloth of any given sieve shall be that given in column 5 (or 6) of Table I within the "Tolerance on Wire Diameter" given in columns 8 and 9. The maximum opening between adjacent parallel wires shall not exceed the nominal width of opening for that sieve by more than the "Tolerance in Maximum Opening" given in column 10 of Table I.

II. WORKMANSHIP

4. The sieves shall be free from imperfections in the sieve cloth or its mounting, as for example, punctured, loose or wavy cloth, imperfections in soldering, etc.

III. MARKING

5. The sieves shall bear a label upon which the manufacturer's nominal opening in metric units (preferably in microns) with the equivalent in inches, together with the size of wire should be given.

NOTE.—It is recommended that, until the new micron designation shall have been thoroughly established, in addition the corresponding United States Standard Sieve Series number be given.

IV. METHODS OF TESTING

Methods of Testing.

6. (*a*) To determine whether the cloth conforms to the requirements of these specifications, the wire diameter shall be measured in each direction by means of a micrometer-microscope or projection apparatus or other suitable apparatus of requisite precision, care being taken in all cases to avoid diffraction effects in measuring to the edge of a wire. Micrometer calipers and steel scales should not be used for this purpose. The mesh shall be determined by counting the number of openings for a distance of at least 6 in. in both directions of the sieve, using a scale, either glass or steel, and a microscope. The use of scales with special rulings for obtaining mesh by counting "interference bands" is recommended in cases where large numbers of any of the sieves with the smaller openings are to be measured. From the wire diameter and the mesh the average opening shall be computed.

NOTE.—No general rule can be given for the number of observations to be taken, except that they be sufficient to cause no change in the result if say 3 or 4 additional measurements are made.

(*b*) Measurements of large openings shall be made by means of the micrometer-microscope or the projection lantern or other suitable apparatus in the same way as wire diameters are measured.

NOTE.—In order to insure the securing of standard sieves it is possible to obtain from the manufacturer or dealer sieves which have been certified by the U. S. Bureau of Standards.

APPENDIX

List of Points to be Considered in Drawing up Specifications for a Sieving Test

I. Apparatus.

1. Enumeration of sieves to be used.
2. Auxiliary apparatus required for collection and reduction of gross sample, sample containers, and apparatus other than the sieves used in making the tests.

II. Sampling.

1. Unit of sampling.
2. Method of collecting representative gross sample.
3. Size of gross sample.
4. Cautions to be taken in handling sample to prevent contamination or change.
5. Reduction of gross sample.

III. Preparation of the Sample for Testing.

1. Drying.
2. Mixing.
3. Special treatment, if any is necessary.
4. Selection of test portion.
5. Size of test portion.

IV. Procedure.

A. Dry Screening:

1. Order in which the sieves are to be used.
2. Detailed description of method of shaking.
3. Detailed description of procedure other than method of shaking.
4. End point.

B. Wet Screening:

1. Liquid to be used.
2. Description of any preliminary separation by classification or other means.
3. Order in which screens are to be used.
4. Detailed description of procedure including amount and method of adding liquid, method of stirring or other agitation.
5. End point.

V. Method of Reporting Results.

VI. Allowable Variations Between Duplicate Tests on the Same Material.

TENTATIVE METHODS OF TENSION TESTING OF METALLIC MATERIALS[1]

Serial Designation: E 8 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924; Revised, 1925.

These methods deal with the form and dimensions, the machining, and the testing of tension test specimens of metallic materials. Such matters as methods of taking samples from which to prepare test specimens are treated in the individual specifications for ferrous metals and for non-ferrous metals.

I. TEST SPECIMENS

1. In recommending the following specimens for use in tension tests it is not intended to exclude entirely the use of other test specimens for special materials or for special forms of material; for example, in tests of tubing and pipe the form of specimen recommended for plate, shape and flat material (Fig. 1) is not generally suitable for use, and a special form of specimen is specified. It is, however, recommended that wherever it is feasible the specimens here recommended be used.

Wire, Rods and Pipe.

2. Tension test specimens of wire are of the full-size diameter as drawn, and tension test specimens of rods of ductile metal are often of the full-size diameter as rolled. Tension test specimens of pipe and of tubing are frequently of full size as manufactured, in which case the ends should be plugged with metal plugs which do not extend within the gage marks.

Plates, Shapes and Flats.

3. The tension test specimen shown in Fig. 1 is recommended for plate, shape and flat material. The thickness of the specimen is that of the material tested.

Note.—When it is desired to use a specimen with a gage length of less than 8 in., the general proportions of Fig. 1 should be followed. Specimens with a gage length of 2 in. are occasionally used. When it is not convenient to machine specimens to the standard shape shown in Fig. 1, specimens may be used with edges machined parallel for the entire length of the specimen. If such specimens are

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing of Metallic Materials, University of Illinois, Urbana, Ill.

These methods are in effect a revision of the Methods for Tension Tests of the Standard Methods of Mechanical Testing of Metallic Materials. The standard methods, which were last published under the Serial Designation: E 1 – 18, have accordingly been withdrawn.

machined with a tool cutting across the edges of the specimen it is important that the transverse tool marks be removed before the specimen is tested.

For thick plate material, it is usually preferable to use a machined specimen rather than to use a specimen the full thickness of the plate.

Up to the present time it has not been possible to fix a definite line between plate material and sheet material.

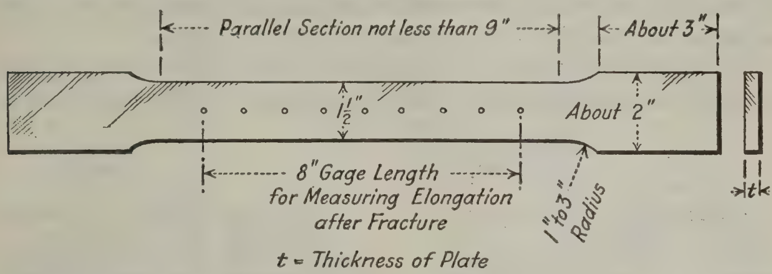


FIG 1

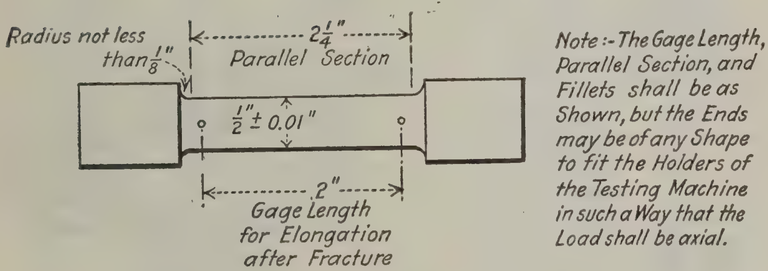
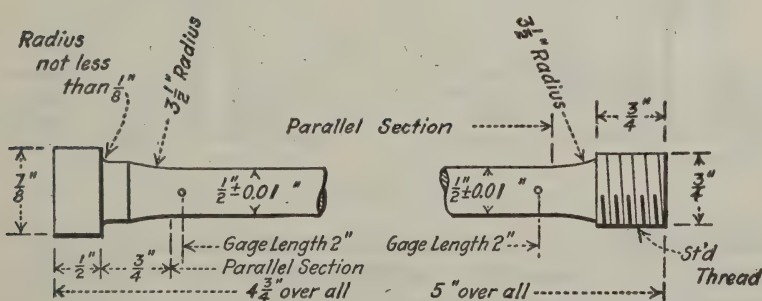


FIG. 2



(*a*) Specimen with shouldered ends. (*b*) Specimen with threaded ends.

FIG. 3

4. The tension test specimen shown in Fig. 2 is recommended for general use in testing metals.[1] The gage length for measuring elongation after fracture is 2 in.

[1] Attention is called to Section 7.

5. When it is necessary to cut specimens from material (other than plate, shape and flat material) which is of such size that the specimen shown in Fig. 2 cannot be used, it is recommended that a specimen with dimensions proportional to those shown in Fig. 2 be used and that the specimen be made as large as feasible. In any such small-size specimen it is especially important that the gage length for measuring elongation be four times the diameter of the specimen.

Measurement of Cross-Section.

6. In all tension tests of metals the actual dimensions of cross-section of the test specimens shall be measured with a micrometer

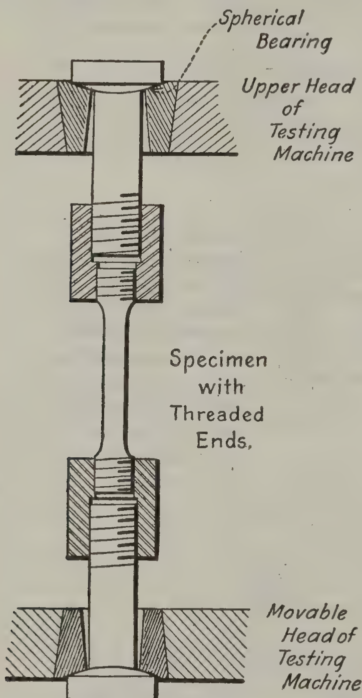


FIG. 4

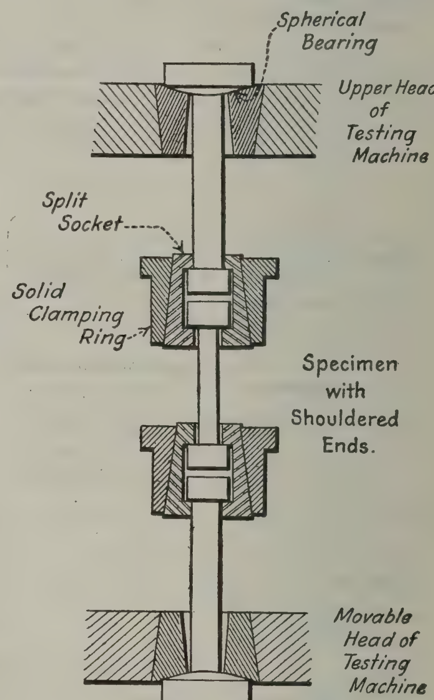


FIG. 5

reading to $\frac{1}{200}$ of the dimension measured, and the stresses shall be computed on the basis of the measured cross-section; it should never be assumed that the dimensions of the measured cross-section are identical with the nominal dimensions.

NOTE.—It will be noted that the tolerance for diameter of specimens shown in Fig. 2 permits the use of specimens 0.505 in. in diameter.

Ends.

7. The shape of the ends of the specimen shown in Fig. 2 are not specified above. For specimens of brittle metal it is desirable

that the diameter of the specimen be changed gradually along its length from the minimum section at the gage length to the diameter at the ends. Fig. 3 shows two forms of end of specimen which have given satisfactory results in tests of brittle metals.

8. Specimens shall be finished so that the surfaces are smooth and free from nicks and tool marks. All ragged edges shall be smoothed. **Finish.**

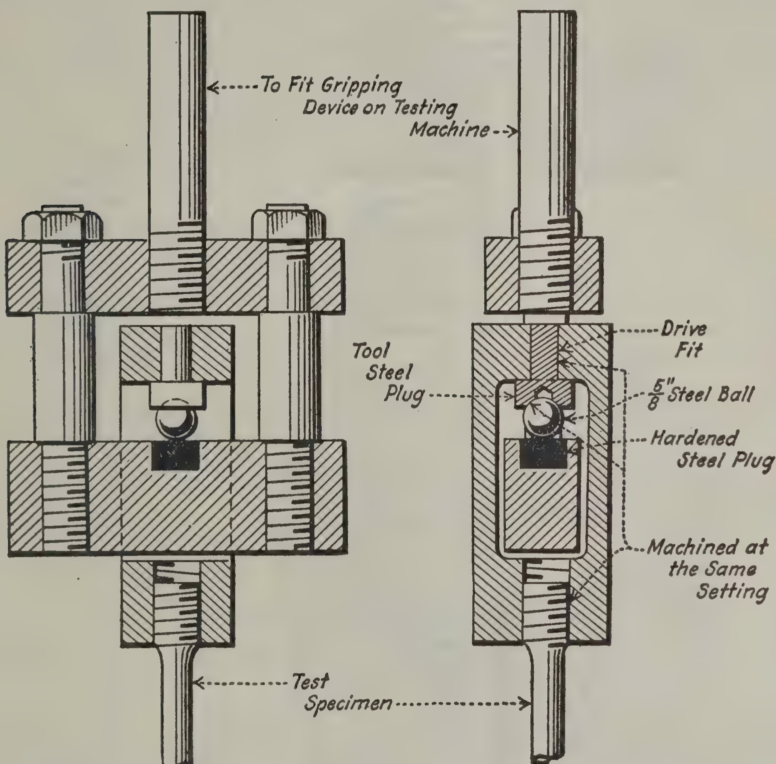


FIG. 6

The gripping device is shown as used with threaded end specimens. A similar device fitted with split sockets would be used with shouldered specimens.

II. GRIPPING DEVICES FOR TENSION TEST SPECIMENS

9. For long bars of ductile metal and for the flat plate test specimen shown in Fig. 1, wedge grips, bearing directly on the end of the test specimen, generally furnish a satisfactory means of gripping the specimens. **Wedge Grips.**

10. For short specimens and for specimens of brittle material, wedge grips are not a satisfactory means of holding test specimens; it is necessary to use machined specimens and to use special means **Grips for Brittle Material.**

for insuring that the specimen, when under load, shall be as nearly as possible in pure axial tension. Fig. 4 shows a device for gripping tension specimens with threaded ends and Fig. 5 shows a device for gripping specimens with shouldered ends. Both these gripping devices are attached to the heads of the testing machine through

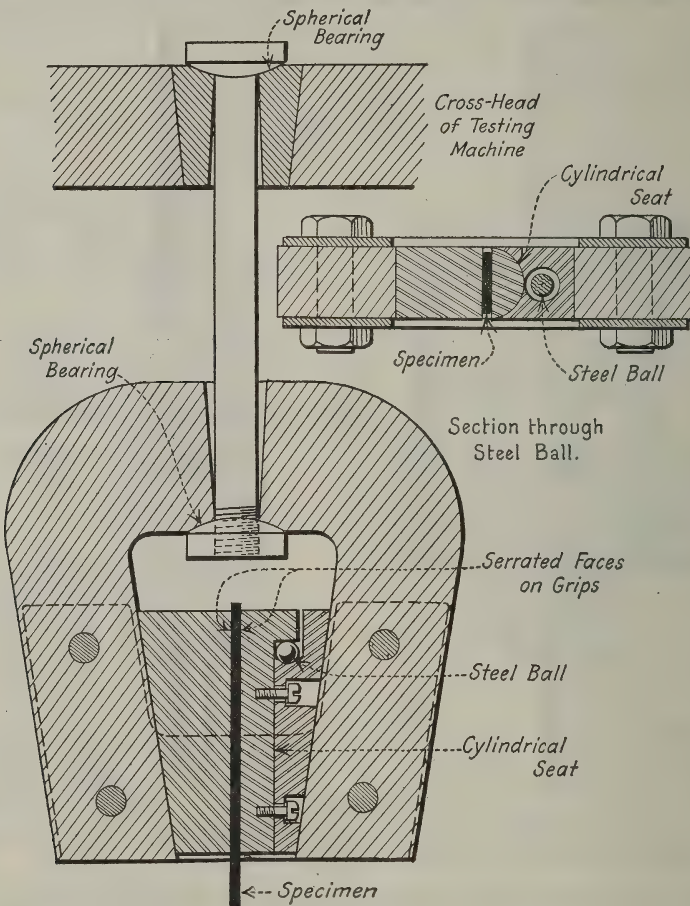


FIG. 7

spherical-seated bearings. The distance between spherical bearings should be as great as is feasible. For brittle materials, even the spherical-seated bearings shown in Figs. 4 and 5 are not always effective in avoiding bending stress on the specimen. The gripping device shown in Fig. 6 has proved satisfactory for testing such speci-

mens, and is an excellent gripping device for any machined tension test specimen.

11. For specimens cut from thin plate material and from sheet material, ordinary wedge grips are liable to cut the specimens near the edges, giving a tearing stress rather than an axial tension. The self-adjusting grips shown in Fig. 7 have proved satisfactory for tests of thin sheet copper and aluminum. **Grips for Sheet Material.**

12. For tests of specimens of wire, ordinary wedge grips are liable to cut the wire at the edges of the grips. This cutting has been **Grips for Wire.**

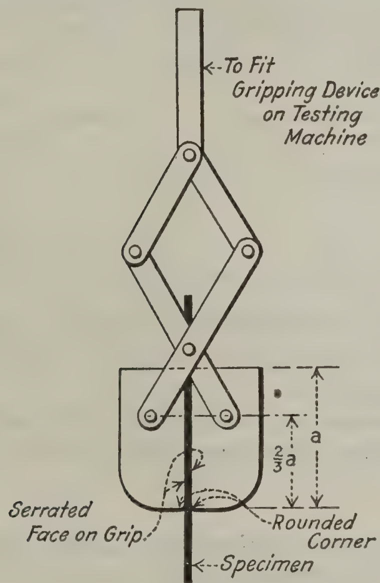


FIG. 8

found to be largely eliminated by the use of grips of the form shown in Fig. 8.

13. In making tension tests, care shall be taken to see that the axis of the test specimen is located in the center line of the heads of the testing machine and that the liners used behind wedge grips are of proper thickness. From time to time the openings in the heads of the testing machine shall be lined up and the heads tested for parallelism. Deviations from correct alignment and from parallelism should not exceed 0.01 in. for any position of the movable head within the range of movement during test. The heads of the machine should be free from sidewise motion that is visible to the unaided eye as the machine is run at its fastest speed. **Alignment.**

III. SPEED OF OPERATION OF TESTING MACHINE

14. The pulling speed has a marked influence on the tensile properties shown by materials tested, an increase in speed increasing the values found for yield point and tensile strength. In testing specimens of metallic materials in gage lengths of 2 and 8 in., the speed of the machine, by which is meant the speed of the cross-head when the machine is running idle, shall conform to the following requirements:

The speed of head of the testing machine shall be such that the load can be accurately weighed, but in no case shall the values given in the following table be exceeded, which values represent the extreme of good laboratory practice for steel:[1]

Specified Minimum Tensile Strength of Material, lb. per sq. in.	Gage Length, in.	Maximum Cross-head Speed, in. per minute	
		Yield Point	Tensile Strength
80 000 or under	2	0.50	2.0
	8	2.00	6.0
Over 80 000	2	0.25	1.0
	8	0.50	2.0

15. In determining the elastic limit in accordance with Method I, Section 17, the cross-head speed for the 2-in. gage length shall not exceed 0.125 in. per minute.

16. In determining the elastic limit by Method II, Section 17, the cross-head speed shall not exceed 0.025 in. per inch of gage length per minute.

IV. THE DETERMINATION OF ELASTIC LIMIT, YIELD POINT, AND TENSILE STRENGTH

Elastic Limit.

17. The term "elastic limit" is defined as follows:

Elastic Limit.—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

The determination of the elastic limit as thus defined would logically involve the application and release of a succession of increasing loads on a test specimen until there was observed deformation after release of a load. This procedure is very slow and since for many materials experience does not indicate any appreciable difference between the elastic limit and the limit of proportionality of stress to strain (sometimes called the proportional limit), the determination of the stress at the limit of proportionality of stress to strain is regarded as an accurate determination of the elastic limit.

[1] The speeds given here are probably too high for satisfactory use in testing non-ferrous metals.

It is obvious that the values obtained in tests for determining the elastic limit will depend on the delicacy of methods and instruments used. It becomes necessary, therefore, that in any test the method used in obtaining the elastic limit be clearly stated. The following methods are in common use for determining a value designated as the elastic limit.

Method I, proposed by Committee A-1 on Steel, and embodied in a number of specifications for steel. (For tension tests only.) The elastic limit shall be determined by an extensometer reading to 0.0002 in. The extensometer shall be attached to the specimen at the gage marks and not to the shoulders of the specimen nor to any part of the testing machine. When the specimen is in place and the extensometer attached, the testing machine shall be operated so as

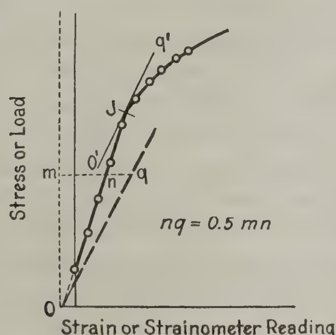


FIG. 9.—Illustrating Method II for Determining Elastic Limit.

to increase the load on the specimen at a uniform rate. The observer shall watch the elongation of the specimen as shown by the extensometer and shall note, for this determination, the load at which the rate of elongation shows a sudden increase. The extensometer may then be removed from the specimen and the test continued to determine the tensile strength.

Method II (proposed by the late J. B. Johnson) is a somewhat more delicate method for locating the elastic limit than is Method I. In Method II, the elastic limit shall be taken as that stress at which the rate of deformation is 50 per cent greater than the initial rate of deformation. A stress-strain diagram is necessary for determining the elastic limit by this method. It is illustrated in Fig. 9. The initial rate of deformation is given by the ratio $mn:Om$. $nq = 0.5\ mn$, $mq = 1.5\ mn$, and the slope of Oq represents a rate of deformation 50 per cent greater than the initial rate. $O'q'$ is drawn parallel to Oq and tangent to the stress-strain diagram. The point of tangency J

locates the elastic limit. In using Method II the same precautions should be observed in attaching the strainometer as for the determination of the elastic limit by Method I. Method II, in tension tests of metals, gives lower values for the elastic limit than does Method I. It is recommended that in using Method II for tension tests the strainometer be of such sensitiveness that it will indicate a change of strain of 0.0001 in. per inch of gage length (0.0001 mm. per millimeter of gage length).

Methods of High Precision.—When it is desired to determine the elastic limit with a high degree of precision, it is suggested that the experimenter devise his own procedure, and in reporting his results describe the procedure in detail, including a statement of the limits of sensitiveness of the apparatus used and of the procedure used in plotting the stress-strain diagram from which the determination of elastic limit is made. It is to be remembered that the accurate determination of the elastic limit requires the use of accurate and sensitive instruments and the use of accurate methods of plotting test data.

Yield Point.

18. The term "yield point" is defined as follows:

Yield Point.—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

Two methods are in use for determining the yield point: (I) The "drop of the beam" method, and (II) the method by use of dividers:

Method I.—In Method I, load is applied to the specimen at a steady rate of increase and the operator keeps the beam in balance by running out the poise at a steady rate. At the yield point the increase of load stops (and for some metals there is an actual falling off of load), but the operator, running out the poise at a steady rate, runs it a trifle beyond the balance position, and the beam of the machine drops for a brief but appreciable interval of time. In a machine fitted with a self-indicating load-measuring device there is a sudden halt of the load-indicating pointer, corresponding to the drop of the beam. The load at the "halt" or the "drop" is recorded, and the corresponding stress is taken as the yield point. This method of determining the yield point requires only one man to conduct the test.

Method II.—In Method II an observer with a pair of dividers watches for visible elongation between two section marks on the specimens. When visible stretch is observed, the load at that instant is noted, and the stress corresponding to the load is taken as the yield point. When a strainometer[1] is used in place of dividers a deforma-

[1] The determination of yield point with a strainometer is sometimes made in tests on non-ferrous metals.

tion of 0.01 in. over a gage length of 2 in. shall be regarded as indicating the yield point.

Some metals do not have a well-defined yield point.

19. The term "tensile strength" is defined as follows: **Tensile Strength.**

Tensile Strength.—The maximum tensile stress which a material is capable of developing.

NOTE.—In practice, it is considered to be the maximum stress carried by a specimen representing the material in a tension test to rupture, under definite prescribed conditions. Tensile strength is computed from the maximum load carried during a tension test and the original cross-sectional area of the specimen.

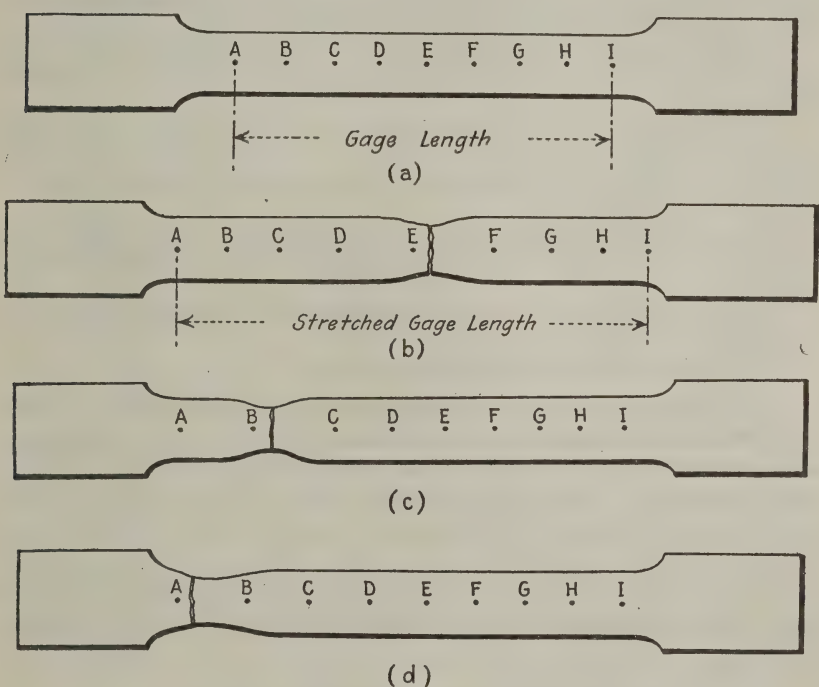


FIG. 10.

The conditions that must be prescribed in a tension test of metallic materials are the form of specimen, the method of gripping the specimen, and the rate of application of load. These are covered in foregoing sections. No further detailed discussion of methods of determining tensile strength is necessary, as there is involved merely the observing and the recording of the maximum load carried by the specimen during the test.

V. THE MEASUREMENT, AFTER FRACTURE, OF THE ELONGATION AND THE REDUCTION OF CROSS-SECTION OF TENSION TEST SPECIMENS

Elongation. 20. The measurement of the elongation after fracture of tension test specimens can be made with sufficient accuracy by means of a pair of dividers and a scale. The elongation should not be reported for any tension test specimen which breaks outside the middle third of the gage length.

NOTE.—If only a few specimens are furnished for testing it is recommended that in marking the gage length for measuring elongation after fracture it be divided into eight parts, and that if a specimen breaks inside the gage length but outside the middle third of the gage length the requirement in the foregoing paragraph be waived, and the elongation be measured and reported as follows:

(1) If the specimen breaks in the middle half of the gage length (between C and G, Figs. 10 (a) and (b)) the elongation is measured directly over the stretched gage length (Fig. 10 (b).

(2) If the specimen breaks between A and C (or between G and I) but nearer B than A (or nearer H than I), as shown in Fig. 10 (c) the elongation from A to C (or from G to I) is measured, and to this is added *twice* the measured elongation from C to F (or from D to G).

(3) If the specimen breaks within one-half a division of an end gage mark (see Fig. 10 (d)) the elongation from A to E (or from E to I) is measured and multiplied by 2.

The above method can be applied to specimens with any gage length, and to turned specimens as well as flat specimens.

This method is not applicable to brass specimens.

Reduction of Area. 21. The measurement of reduction of the dimensions of the cross-section of a tension test specimen may be made by the direct measurement with a micrometer of the smallest section of the fractured specimen. For round specimens this measurement can usually best be made by holding the broken pieces together in a vise or between centers and then measuring the average diameter of the smallest cross-section by means of a micrometer fitted with points so shaped that they will come in contact with the specimen at its smallest diameter.

VI. PLOTTING STRESS-STRAIN DIAGRAMS

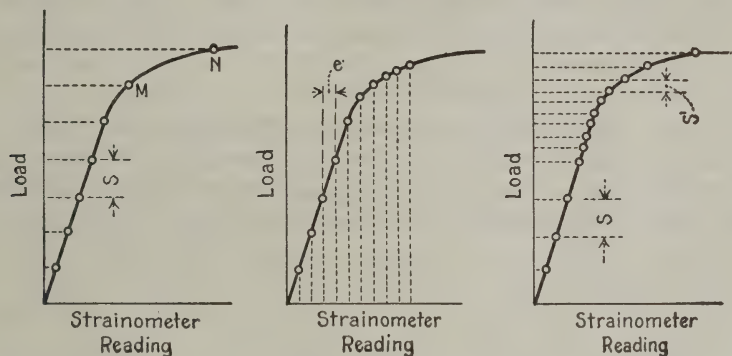
22. A stress-strain diagram is a diagram plotted with values of stress as ordinates and values of strain as abscissas.

NOTE.—The use of the term "stress-strain diagram" is frequently extended to cover diagrams plotted with values of applied load, or applied moment, as ordinates and with values of stretch, compression, deflection, or twist as abscissas.

Stress-strain diagrams are in some cases drawn directly by an autographic attachment to the testing machine. A more usual method of procedure consists in taking a series of load readings (from the

balanced scale beam of the testing machine) with corresponding readings of the strain-indicating apparatus. The term "strainometer reading" will be used to denote the reading of the strain-measuring instrument. From these readings, or from values computed from them, there is plotted a diagram with stress-indicating values (load or stress) as ordinates, and strain-indicating values (elongation or strain) as abscissas. In planning such a test it is necessary to decide on the increment of load or the increment of reading of strainometer to be used between successive readings.

In Fig. 11 are shown three typical stress-strain diagrams. The material for all three tests is the same, and the test specimens are all of the same size, so that the diagrams should be the same. The



(*a*) With Equal Increments of Stress. (*b*) With Equal Increments of Strain. (*c*) With Two Values of Increments of Stress.

FIG. 11.—Illustrating Three Methods of Plotting Load-Deformation Curves.

diagram shown in Fig. 11 (*a*) is plotted from points determined by taking increments of load (S). In this diagram it is seen that owing to the shape of the curve, data for locating points between M and N are lacking. That particular portion of the curve (the "knee" of the curve) is the part for which it is especially desirable to locate several points on the diagram.

In Fig. 11 (*b*) is shown a diagram plotted from points determined by taking increments of strainometer reading (e). It will be noted that for this diagram there are located several points near the knee of the curve, and the shape of the diagram in this important region is much more definitely determined than for the curve shown in Fig. 11 (*a*).

The custom of choosing increments of load rather than increments of strainometer reading is quite common in tests of materials and is

followed because, in general, it is easier to compute increments of load than it is to compute increments of strainometer reading. An estimate of the load necessary to stress the specimen up to the knee of the curve is made and some fraction (frequently one-tenth) of this value is taken as an increment. The computation of the corresponding increment of strainometer reading is more complicated.

Sometimes the practice is followed of applying a few increments of load as determined above, and then applying load in much smaller increments (S') until the knee of the curve is passed. Fig. 11 (*c*) shows a diagram obtained in this manner. This method involves a marked increase in the number of readings necessary for a test and with unknown material there is always some danger that the knee of the curve will be reached before the use of small increments of load is begun.

The following method of choosing increments for a test is suggested: Estimate the load corresponding to the knee of the stress-strain diagram and choose a value for increment of load about one-tenth of this value. Apply this increment of load (S) once and note the corresponding change of reading for the strainometer (e). Then for the remainder of the test, use for the increment of strainometer reading a value which corresponds to some convenient interval on the scale of the strainometer, and which is approximately equal to (e).

TENTATIVE METHODS OF COMPRESSION TESTING OF METALLIC MATERIALS[1]

Serial Designation: E 9 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924; Revised, 1925.

These methods deal with the form and dimensions, the machining, and the testing of compression test specimens of metallic materials. Such matters as methods of taking samples from which to prepare test specimens are treated in the individual specifications for ferrous metals and for non-ferrous metals.

I. TEST SPECIMENS

1. In recommending the following specimens for use in compression tests it is not intended to exclude entirely the use of other test specimens for special materials or for special forms of material. It is, however, recommended that wherever it is feasible the specimens here recommended be used.

2. It is recommended that standard compression specimens be in the form of circular cylinders. The ends of a specimen should be plane, as nearly as can be determined by the use of a straight edge and the unaided eye. In most cases this requirement necessitates the turning or the grinding of the ends of the specimen.

3. There are recognized three classes of compression specimens for metallic materials, designated as short specimens, medium-length specimens, and long specimens, respectively. Short specimens are used for compression tests of such metals as bearing metals, which in service are used in the form of a thin plate or shell to carry load perpendicular to the surface. Medium-length specimens are used for determining the general compressive strength properties of metallic materials. Long specimens are best adapted for determining the modulus of elasticity in compression of metallic materials. In reporting the results of a compression test, it is important that the dimensions of the test specimen be given.

[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing of Metallic Materials, University of Illinois, Urbana, Ill.

These methods are in effect a revision of the Methods for Compression Tests of the Standard Methods of Mechanical Testing of Metallic Materials. The standard methods, which were last published under the Serial Designation: E 1 – 18, have accordingly been withdrawn.

4. Fig. 1 shows the proportions of the three classes of compression specimens. Suggested dimensions for compression specimens for general use are:

Short specimens: $d = 1\frac{1}{8}$ in. ± 0.01 in. (area of cross-section is approximately 1 sq. in.), $h = 1$ in.;

Medium-length specimens: $d = 0.798$ in. ± 0.01 in., $h = 2\frac{3}{8}$ in., or
$d = 1$ in. ± 0.01 in., $h = 3$ in., or
$d = 1\frac{1}{8}$ in. ± 0.01 in., $h = 3\frac{3}{8}$ in.

Long specimens: $d = 1\frac{1}{4}$ in. ± 0.01 in., $h = 12\frac{1}{2}$ in.

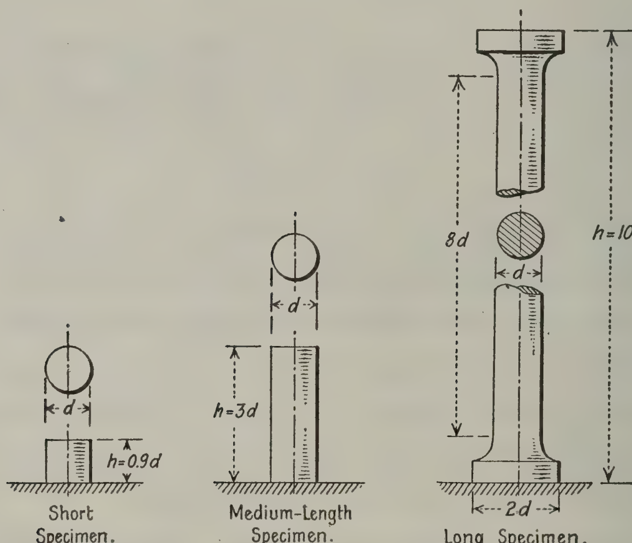


FIG. 1.

5. In all compression tests of metallic materials, the actual dimensions of cross-section of the test specimens shall be measured with a micrometer reading to $\frac{1}{200}$ of the dimension measured, and stresses shall be computed on the basis of the measured cross-section. It should never be assumed that the dimensions of the cross-section of the specimen are identical with the nominal dimensions.

6. Specimens shall be finished so that the surfaces are smooth and free from nicks and tool marks. All ragged edges shall be smoothed.

II. BEARING BLOCKS FOR COMPRESSION TEST SPECIMENS

7. Both ends of a compression test specimen shall bear on a carefully machined plane surface. The bearing blocks shall either be made of hardened steel, or shall be faced with hardened steel.

8. One end of a compression test specimen shall bear on a spherical-seated compression block.[1] Fig. 2 shows a satisfactory arrangement of compression specimens and bearing blocks. It is desirable that the spherical-seated bearing block be at the upper end of the test specimen (for specimens tested with the axis vertical). It is important that the center of the spherical surface of the spherical-seated bearing block should be in the flat face which bears on the specimen, otherwise lack of parallelism between faces of the specimen sets up a bending moment in the specimen.[2]

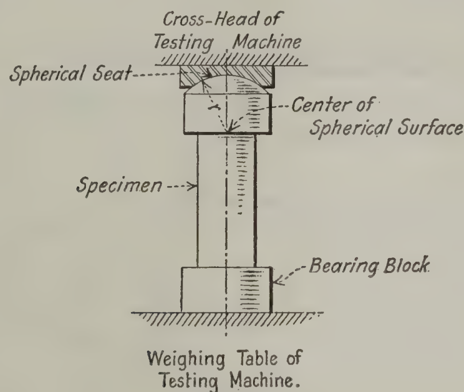


FIG. 2.

9. It should be borne in mind that the object of a spherical-seated bearing block is to give the specimen as even a distribution of initial load as is possible. Owing to friction, the spherical-seated bearing cannot be relied on to adjust itself to bending action which may occur during the test.

10. In making compression tests, care should be taken to preserve the alignment of the heads of the testing machine, and to center the specimen in the testing machine. From time to time the heads of the testing machine shall be tested for parallelism. Deviations from parallelism should not exceed 0.01 in. The movable head of the machine should be free from sidewise motion that is visible to the unaided eye as the machine is run at its fastest speed.

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[1] The device shown in Fig. 3 has been successfully used in compression tests of metallic specimens whose ends were machined with extreme care. It will be noted that this device does not involve a spherical seated compression block bearing directly on the test specimen. This device or one similar has been successfully used in the British National Physical Laboratory and in the laboratories of the U. S. Air Service at McCook Field, Dayton, Ohio.

[2] See Mont Schuyler, "Spherical Bearings," *Proceedings*, Am. Soc. Testing Mats., Vol. XIII, p. 1004 (1913).

III. SPEED OF OPERATION OF TESTING MACHINE

11. In making compression tests of specimens of metallic materials the speed of the machine, by which is meant the speed of the cross-head when the machine is running idle, shall not be greater than the values given in the following table:

LENGTH OF SPECIMEN, IN.	MAXIMUM CROSS-HEAD SPEED, IN. PER MINUTE
1.00	0.05
3.00 or greater	0.10

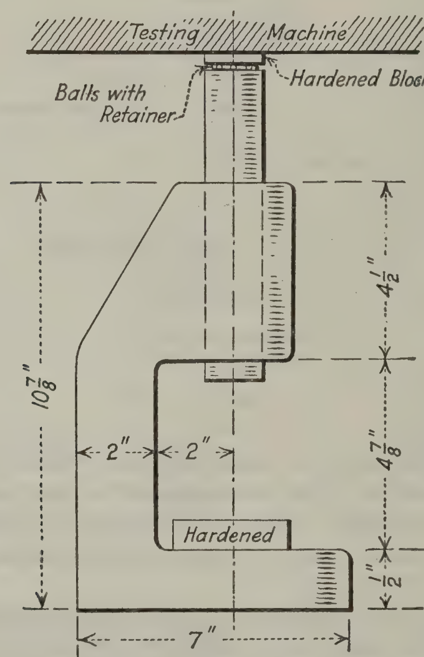

FIG. 3.—Compression Tool.

12. In making tests to determine the elastic limit in compression of metallic materials, the cross-head speed of the testing machine shall in no case exceed the allowable speed of head for the corresponding tension test of the material, as given in Sections 15 and 16 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 – 25 T) of the American Society for Testing Materials.[1]

[1] See p. 770.

IV. THE DETERMINATION OF ELASTIC LIMIT, YIELD POINT, AND COMPRESSIVE STRENGTH

13. (*a*) The term "elastic limit" in compression is defined the same as in tension, as follows:

> *Elastic Limit.*—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

(*b*) The same general methods for determining elastic limit, as given in Section 17 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 – 25 T) of the American Society for Testing Materials,[1] shall be used. For compression tests, Method II or a still more precise method seems generally suitable.

14. (*a*). The term "yield point" in compression is defined the same as in tension, as follows:

> *Yield Point.*—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

(*b*) The same methods for determining yield point as given in Section 18 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 – 25 T)[1] shall be used. The method by the use of dividers or by the use of a strainometer seems generally suitable for compression testing.

15. The term "compressive strength" is defined as follows:

> *Compressive Strength.*—The maximum compressive stress which a material is capable of developing.

NOTE.—In the case of a material which fails in compression by a shattering fracture the compressive strength has a very definite value. In the case of materials which do not fail in compression by a shattering fracture the value obtained for compressive strength is an arbitrary value depending upon the degree of distortion which is regarded as indicating complete failure of the metal.

V. PLOTTING STRESS-STRAIN DIAGRAMS

16. The discussion and directions given in Section 22 of the Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 – 25 T)[1] apply to plotting stress-strain diagrams for compression tests.

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[1] See p. 770.

TENTATIVE METHODS OF BRINELL HARDNESS TESTING OF METALLIC MATERIALS[1]

Serial Designation: E 10 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1924; REVISED, 1925.

The Brinell test for determining the hardness of metallic materials consists in applying a known load to the surface of the material to be tested through a hardened steel ball of known diameter. The diameter (or depth) of the resulting permanent impression in the metal is measured. The Brinell hardness number is taken as the quotient of the applied load divided by the area of the surface of the impression, which is assumed to be spherical. If P is the applied load (measured in kilograms), D is the diameter of the steel ball (measured in millimeters) and d is the diameter of the impression (measured in millimeters), then

$$\text{B.H.N.} = \frac{P}{\frac{\pi D}{2}\left(D - \sqrt{D^2 - d^2}\right)}$$

in which B.H.N. is the Brinell hardness number in kilograms per square millimeter.

If the steel ball were not deformed under the applied load and if the impression were truly spherical, then the above formula would be a general one, and any combination of applied load and size of ball could be used. As the impression is not quite a spherical surface (since there must always be some deformation of the steel ball and some recovery of form of the metal in the impression), for a standard Brinell test the size and characteristics of the ball and the magnitude of the applied load must be standardized. (A standard ball 10 mm. in diameter and a load of 3000 kg. for hard metals and 500 kg. for soft metals is standard practice. See Sections 3 and 6.

In Table I is given the Brinell hardness number corresponding to various diameters of impression for both 500 and 3000 kg. load, making it unnecessary to compute for each test the value of the Brinell Hardness Number by the above formula.

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[1] Criticisms of these Tentative Methods are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing of Metallic Materials, University of Illinois, Urbana, Ill.

These methods are in effect a revision of the Methods for Brinell Hardness Tests of the Standard Methods of Mechanical Testing of Metallic Materials. The standard methods, which were last published under the Serial Designation: E 1 – 18, have accordingly been withdrawn.

I. APPARATUS

1. The divisions on the micrometer scale of the microscope or other measuring device used for the measurement of the diameter of impression shall be such as to permit the direct measuring of the diameter to 0.1 mm. and the estimation of the diameter to 0.01 mm. **Micrometer Microscope.**

2. (*a*) The balls used in Brinell hardness testing shall be of hardened steel or other equally hard material. **Brinell Ball.**

(*b*) A ball to be suitable for use shall not show a permanent change in diameter greater than 0.0025 mm. (0.0001 in.) when pressed with a force of 3000 kg. against a piece of steel having a Brinell hardness number of 500 or greater.[1]

(*c*) If a ball is used in a test of a specimen which shows a Brinell hardness number greater than 500, the ball shall be remeasured after the test. Should the ball show a permanent change in diameter greater than that permitted in Paragraph (*b*), the ball shall be unsuitable for further use in a standard test.[1]

3. The standard ball for Brinell hardness testing shall be 10 mm. (0.3937 in.) in diameter with a deviation from this value of not more than 0.01 mm. (0.0004 in.) in any diameter, but the diameter of any ball measured at various points shall be constant within a tolerance of ±0.0025 mm. (0.0001 in.). **Size of Standard Ball.**

For testing very small specimens or very thin specimens it is sometimes necessary to make Brinell hardness tests with a ball less than 10 mm. in diameter. For such tests (which are not to be regarded as standard Brinell tests) the relation between applied load, P, measured in kilograms, and diameter of ball, D, measured in millimeters, should be

$$P = 3000 \frac{D^2}{10^2} = 30 D^2 \text{ for iron and steel}$$

$$\text{and } P = 500 \frac{D^2}{10^2} = 5 D^2 \text{ for brass, bronze and soft metals in general.}$$

For extremely soft metals the relation $P = D^2$ may be used.

Balls differing in size from the standard 10-mm. ball shall conform to the requirements for the material and the tolerance in size of diameter as specified for the standard ball.

II. TEST SPECIMENS

Specimens used in Brinell hardness testing vary greatly in form since it is frequently desirable to make the impression upon a part to be used in the finished product rather than upon a sample test specimen.

4. The thickness of the piece tested shall be such that no bulge or other marking showing the effect of the load appears on the side of **Thickness of Specimen.**

[1] In testing softer metals, it is not, of course, necessary to have balls of this extreme hardness. However, a ball to be suitable for use should withstand pressure against the hardest material to be tested without showing a permanent change in diameter greater than 0.0025 mm.

TABLE I.—BRINELL HARDNESS NUMBERS.

Steel Ball, 10 mm. in Diameter, Pressures of 500 and 3000 kg.
Prepared by the Engineering Mechanics Section, Bureau of Standards.

The values given in this table for hardness numbers are merely solutions of the equation given in the introductory paragraphs. They do not imply that Brinell tests are feasible on materials of a hardness indicated by the highest values in the table.

Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number	
	500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load
2.00	158	945	2.50	100	601	3.00	69.1	415	3.50	50.3	302	4.00	38.1	229
2.01	156	936	2.51	99.4	597	3.01	68.6	412	3.51	50.0	300	4.01	37.9	228
2.02	154	926	2.52	98.6	592	3.02	68.2	409	3.52	49.7	298	4.02	37.7	226
2.03	153	917	2.53	97.8	587	3.03	67.7	406	3.53	49.4	297	4.03	37.5	225
2.04	151	908	2.54	97.1	582	3.04	67.3	404	3.54	49.2	295	4.04	37.3	224
2.05	150	899	2.55	96.3	578	3.05	66.8	401	3.55	48.9	293	4.05	37.1	223
2.06	148	890	2.56	95.5	573	3.06	66.4	398	3.56	48.6	292	4.06	37.0	222
2.07	147	882	2.57	94.8	569	3.07	65.9	395	3.57	48.3	290	4.07	36.8	221
2.08	146	873	2.58	94.0	564	3.08	65.5	393	3.58	48.0	288	4.08	36.6	219
2.09	144	865	2.59	93.3	560	3.09	65.0	390	3.59	47.7	286	4.09	36.4	218
2.10	143	856	2.60	92.6	555	3.10	64.6	388	3.60	47.5	285	4.10	36.2	217
2.11	141	848	2.61	91.8	551	3.11	64.2	385	3.61	47.2	283	4.11	36.0	216
2.12	140	840	2.62	91.1	547	3.12	63.8	383	3.62	46.9	282	4.12	35.8	215
2.13	139	832	2.63	90.4	543	3.13	63.3	380	3.63	46.7	280	4.13	35.7	214
2.14	137	824	2.64	89.7	538	3.14	62.9	378	3.64	46.4	278	4.14	35.5	213
2.15	136	817	2.65	89.0	534	3.15	62.5	375	3.65	46.1	277	4.15	35.3	212
2.16	135	809	2.66	88.4	530	3.16	62.1	373	3.66	45.9	275	4.16	35.1	211
2.17	134	802	2.67	87.7	526	3.17	61.7	370	3.67	45.6	274	4.17	34.9	210
2.18	132	794	2.68	87.0	522	3.18	61.3	368	3.68	45.4	272	4.18	34.8	209
2.19	131	787	2.69	86.4	518	3.19	60.9	366	3.69	45.1	271	4.19	34.6	208
2.20	130	780	2.70	85.7	514	3.20	60.5	363	3.70	44.9	269	4.20	34.4	207
2.21	129	772	2.71	85.1	510	3.21	60.1	361	3.71	44.6	268	4.21	34.2	205
2.22	128	765	2.72	84.4	507	3.22	59.8	359	3.72	44.4	266	4.22	34.1	204
2.23	126	758	2.73	83.8	503	3.23	59.4	356	3.73	44.1	265	4.23	33.9	203
2.24	125	752	2.74	83.2	499	3.24	59.0	354	3.74	43.9	263	4.24	33.7	202
2.25	124	745	2.75	82.6	495	3.25	58.6	352	3.75	43.6	262	4.25	33.6	201
2.26	123	738	2.76	81.9	492	3.26	58.3	350	3.76	43.4	260	4.26	33.4	200
2.27	122	732	2.77	81.3	488	3.27	57.9	347	3.77	43.1	259	4.27	33.2	199
2.28	121	725	2.78	80.8	485	3.28	57.5	345	3.78	42.9	257	4.28	33.1	198
2.29	120	719	2.79	80.2	481	3.29	57.2	343	3.79	42.7	256	4.29	32.9	198
2.30	119	712	2.80	79.6	477	3.30	56.8	341	3.80	42.4	255	4.30	32.8	197
2.31	118	706	2.81	79.0	474	3.31	56.5	339	3.81	42.2	253	4.31	32.6	196
2.32	117	700	2.82	78.4	471	3.32	56.1	337	3.82	42.0	252	4.32	32.4	195
2.33	116	694	2.83	77.9	467	3.33	55.8	335	3.83	41.7	250	4.33	32.3	194
2.34	115	688	2.84	77.3	464	3.34	55.4	333	3.84	41.5	249	4.34	32.1	193
2.35	114	682	2.85	76.8	461	3.35	55.1	331	3.85	41.3	248	4.35	32.0	192
2.36	113	676	2.86	76.2	457	3.36	54.8	329	3.86	41.1	246	4.36	31.8	191
2.37	112	670	2.87	75.7	454	3.37	54.4	326	3.87	40.9	245	4.37	31.7	190
2.38	111	665	2.88	75.1	451	3.38	54.1	325	3.88	40.6	244	4.38	31.5	189
2.39	110	659	2.89	74.6	448	3.39	53.8	323	3.89	40.4	242	4.39	31.4	188
2.40	109	653	2.90	74.1	444	3.40	53.4	321	3.90	40.2	241	4.40	31.2	187
2.41	108	648	2.91	73.6	441	3.41	53.1	319	3.91	40.0	240	4.41	31.1	186
2.42	107	643	2.92	73.0	438	3.42	52.8	317	3.92	39.8	239	4.42	30.9	185
2.43	106	637	2.93	72.5	435	3.43	52.5	315	3.93	39.6	237	4.43	30.8	185
2.44	105	632	2.94	72.0	432	3.44	52.2	313	3.94	39.4	236	4.44	30.6	184
2.45	104	627	2.95	71.5	429	3.45	51.8	311	3.95	39.1	235	4.45	30.5	183
2.46	104	621	2.96	71.0	426	3.46	51.5	309	3.96	38.9	234	4.46	30.3	182
2.47	103	616	2.97	70.5	423	3.47	51.2	307	3.97	38.7	232	4.47	30.2	181
2.48	102	611	2.98	70.1	420	3.48	50.9	306	3.98	38.5	231	4.48	30.0	180
2.49	101	606	2.99	69.6	417	3.49	50.6	304	3.99	38.3	230	4.49	29.9	179

TABLE I—(*Continued*).

Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number		Diameter of Indentation, mm.	Brinell Hardness Number	
	500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load		500-kg. Load	3000-kg. Load
4.50	29.8	179	5.00	23.8	143	5.50	19.3	116	6.00	15.9	95.5	6.50	13.3	79.6
4.51	29.6	178	5.01	23.7	142	5.51	19.2	115	6.01	15.9	95.1	6.51	13.2	79.3
4.52	29.5	177	5.02	23.6	141	5.52	19.2	115	6.02	15.8	94.8	6.52	13.2	79.0
4.53	29.3	176	5.03	23.5	141	5.53	19.1	114	6.03	15.7	94.4	6.53	13.1	78.7
4.54	29.2	175	5.04	23.4	140	5.54	19.0	114	6.04	15.7	94.1	6.54	13.1	78.4
4.55	29.1	174	5.05	23.3	140	5.55	18.9	114	6.05	15.6	93.7	6.55	13.0	78.2
4.56	28.9	174	5.06	23.2	139	5.56	18.9	113	6.06	15.6	93.4	6.56	13.0	77.9
4.57	28.8	173	5.07	23.1	138	5.57	18.8	113	6.07	15.5	93.0	6.57	12.9	77.6
4.58	28.7	172	5.08	23.0	138	5.58	18.7	112	6.08	15.4	92.7	6.58	12.9	77.3
4.59	28.5	171	5.09	22.9	137	5.59	18.6	112	6.09	15.4	92.3	6.59	12.8	77.1
4.60	28.4	170	5.10	22.8	137	5.60	18.6	111	6.10	15.3	92.0	6.60	12.8	76.8
4.61	28.3	170	5.11	22.7	136	5.61	18.5	111	6.11	15.3	91.7	6.61	12.8	76.5
4.62	28.1	169	5.12	22.6	135	5.62	18.4	110	6.12	15.2	91.3	6.62	12.7	76.2
4.63	28.0	168	5.13	22.5	135	5.63	18.3	110	6.13	15.2	91.0	6.63	12.7	76.0
4.64	27.9	167	5.14	22.4	134	5.64	18.3	110	6.14	15.1	90.6	6.64	12.6	75.7
4.65	27.8	167	5.15	22.3	134	5.65	18.2	109	6.15	15.1	90.3	6.65	12.6	75.4
4.66	27.6	166	5.16	22.2	133	5.66	18.1	109	6.16	15.0	90.0	6.66	12.5	75.2
4.67	27.5	165	5.17	22.1	133	5.67	18.1	108	6.17	14.9	89.6	6.67	12.5	74.9
4.68	27.4	164	5.18	22.0	132	5.68	18.0	108	6.18	14.9	89.3	6.68	12.4	74.7
4.69	27.3	164	5.19	21.9	132	5.69	17.9	107	6.19	14.8	89.0	6.69	12.4	74.4
4.70	27.1	163	5.20	21.8	131	5.70	17.8	107	6.20	14.8	88.7	6.70	12.4	74.1
4.71	27.0	162	5.21	21.7	130	5.71	17.8	107	6.21	14.7	88.3	6.71	12.3	73.9
4.72	26.9	161	5.22	21.6	130	5.72	17.7	106	6.22	14.7	88.0	6.72	12.3	73.6
4.73	26.8	161	5.23	21.6	129	5.73	17.6	106	6.23	14.6	87.7	6.73	12.2	73.4
4.74	26.6	160	5.24	21.5	129	5.74	17.6	105	6.24	14.6	87.4	6.74	12.2	73.1
4.75	26.5	159	5.25	21.4	128	5.75	17.5	105	6.25	14.5	87.1	6.75	12.1	72.8
4.76	26.4	158	5.26	21.3	128	5.76	17.4	105	6.26	14.5	86.7	6.76	12.1	72.6
4.77	26.3	158	5.27	21.2	127	5.77	17.4	104	6.27	14.4	86.4	6.77	12.1	72.3
4.78	26.2	157	5.28	21.1	127	5.78	17.3	104	6.28	14.4	86.1	6.78	12.0	72.1
4.79	26.1	156	5.29	21.0	126	5.79	17.2	103	6.29	14.3	85.8	6.79	12.0	71.8
4.80	25.9	156	5.30	20.9	126	5.80	17.2	103	6.30	14.2	85.5	6.80	11.9	71.6
4.81	25.8	155	5.31	20.9	125	5.81	17.1	103	6.31	14.2	85.2	6.81	11.9	71.3
4.82	25.7	154	5.32	20.8	125	5.82	17.0	102	6.32	14.1	84.9	6.82	11.8	71.1
4.83	25.6	154	5.33	20.7	124	5.83	17.0	102	6.33	14.1	84.6	6.83	11.8	70.8
4.84	25.5	153	5.34	20.6	124	5.84	16.9	101	6.34	14.0	84.3	6.84	11.8	70.6
4.85	25.4	152	5.35	20.5	123	5.85	16.8	101	6.35	14.0	84.0	6.85	11.7	70.4
4.86	25.3	152	5.36	20.4	123	5.86	16.8	101	6.36	13.9	83.7	6.86	11.7	70.1
4.87	25.1	151	5.37	20.3	122	5.87	16.7	100	6.37	13.9	83.4	6.87	11.6	69.9
4.88	25.0	150	5.38	20.3	122	5.88	16.7	99.9	6.38	13.8	83.1	6.88	11.6	69.6
4.89	24.9	150	5.39	20.2	121	5.89	16.6	99.5	6.39	13.8	82.8	6.89	11.6	69.4
4.90	24.8	149	5.40	20.1	121	5.90	16.5	99.2	6.40	13.7	82.5	6.90	11.5	69.2
4.91	24.7	148	5.41	20.0	120	5.91	16.5	98.8	6.41	13.7	82.2	6.91	11.5	68.9
4.92	24.6	148	5.42	19.9	120	5.92	16.4	98.4	6.42	13.6	81.9	6.92	11.4	68.7
4.93	24.5	147	5.43	19.9	119	5.93	16.3	98.0	6.43	13.6	81.6	6.93	11.4	68.4
4.94	24.4	146	5.44	19.8	119	5.94	16.3	97.7	6.44	13.5	81.3	6.94	11.4	68.2
4.95	24.3	146	5.45	19.7	118	5.95	16.2	97.3	6.45	13.5	81.0	6.95	11.3	68.0
4.96	24.2	145	5.46	19.6	118	5.96	16.2	96.9	6.46	13.4	80.7	6.96	11.3	67.7
4.97	24.1	144	5.47	19.5	117	5.97	16.1	96.6	6.47	13.4	80.4	6.97	11.3	67.5
4.98	24.0	144	5.48	19.5	117	5.98	16.0	96.2	6.48	13.4	80.1	6.98	11.2	67.3
4.99	23.9	143	5.49	19.4	116	5.99	16.0	95.9	6.49	13.3	79.8	6.99	11.2	67.0

the piece opposite the impression. In any event the thickness of the specimen shall be at least 10 times the depth of impression.

Finish of Specimen. 5. The surface on which the impression is to be made shall be filed, ground, machined, or polished with emery paper so that the edge of the impression shall be clearly enough defined to permit of the measurement of the diameter within 0.01 mm. (0.0004 in.).

III. APPLICATION AND MAGNITUDE OF LOAD

Magnitude. 6. The load in the standard Brinell test shall be 3000 kg. for iron and steel and 500 kg. for brass, bronze and soft metals generally.

NOTE.—For extremely soft metals a load of 100 kg. is sometimes used. If for any reason it is necessary to use loads differing from those specified above, the load used should be specifically stated in the test report, conveniently as a subscript of the hardness numeral.

Application. 7. (*a*) The load shall be applied steadily to the specimen without jerk.

(*b*) The load shall be applied for at least 10 seconds in the case of iron and steel and for at least 30 seconds in the case of other metals.[1]

(*c*) The center line of the applied load shall be far enough from any edge of the specimen so that when a straight-edge is applied to the edge after the load has been applied, the eye can detect no bulge in the edge due to the load and the resulting impression.

IV. MEASUREMENT OF IMPRESSION

8. In the standard Brinell hardness test the diameter of the impression shall be measured.

Measurement of Diameter of Impression. 9. When a micrometer microscope is used to measure the diameter of the impression the sharpness of definition of the edge of the impression can be increased by the use of a movable lamp for illuminating the specimen, placing the lamp so that the contrast of light and shade will bring first one edge of the impression, then the other into sharp definition. In testing very hard material, the sharpness of definition of the impression can be somewhat increased by the use of a ball lightly etched with nitric acid, or by the use of some pigment, such as Prussian blue, on the ball. In testing material in which there is considerable recovery of shape, the material may first be coated with a dull black pigment, such as drawing ink or a mixture of graphite and alcohol. The edge of the impression is rendered clear on a surface so coated.

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[1] It was suggested in discussion that for magnesium and magnesium alloys the minimum time of application be 2 minutes. It was also suggested that for any given soft metal a definite time for holding the load be specified rather than a minimum time. See discussion of the Report of Committee E-1, *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part 1, pp. 729 and 730 (1924).

BRINELL HARDNESS TESTS IN WHICH DEPTH OF IMPRESSION IS MEASURED IN PLACE OF DIAMETER

When it is necessary to make Brinell hardness tests very rapidly, as is sometimes the case when the Brinell test is used as a control test for the output of a heat-treating furnace, the depth of impression under load, or after the release of load, can be measured more expeditiously than the diameter of impression. The depth of impression is usually determined from the relative motion of the plunger carrying the ball and the specimen. This method of determining Brinell hardness is not to be regarded as a standard method, and when requirements of speed necessitate its use, the Brinell hardness number should not be determined from the geometrical relation of depth of impression to diameter, based on the assumption that the impression has a spherical surface. In such tests the relation between the Brinell hardness number and depth of impression may be determined with a fair degree of accuracy from tests on specimens of material of different degrees of hardness, several impressions being made on each specimen, and the depth under load, or the depth on release of load, and the diameter of impression after release of load being measured for each impression. By the use of a diagram or by interpolation, a table giving the Brinell hardness number for a given depth of impression can be prepared.

V. CALIBRATION OF BRINELL HARDNESS TESTING APPARATUS

10. A load-measuring device which is in common use for Brinell hardness testing machines involves the measurement of fluid pressure on a piston of known diameter, working in a cylinder with so perfect a fit that packing is not necessary. To this piston is attached the ball. Other load-measuring devices use combinations of weights and levers. The use of dead weights acting on a small piston is to be preferred to the ordinary Bourdon gage for measuring the intensity of fluid pressure.

11. The load-measuring device of a Brinell hardness testing machine may be calibrated by the use of dead weights, or by the use of dead weights and proving levers or by an elastic calibration bar or spring, in the manner described in the Standard Methods of Verification of Testing Machines (Serial Designation: E 4) of the American Society for Testing Materials.[1]

12. The load-measuring device of a Brinell hardness testing machine may be calibrated by making a series of impressions on specimens of different degrees of hardness, measuring the diameters of the impressions, making a second series of impressions by the use of any standardized testing machine and standard steel ball, and, using the same measuring device as for the first series of impressions, measuring the diameters of this second series of impressions. The error of the machine under calibration can be determined by the relative average

[1] 1924 Book of A.S.T.M. Standards. See also J. L. Jones and C. H. Marshall, "A New Method for Calibrating Brinell Hardness Testing Machines," *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part II, p. 392 (1920).

values of the hardness numbers determined by its use and by the use of the standardized testing machine.

13. When the above companion-impression method of calibrating a Brinell hardness testing machine is used it is recommended that the specimens used be approximately 1 by 1 by 12 in. in size, and that specimens with as wide a range of hardness as is feasible be used. It is recommended that impressions made by the machine under calibration be spaced about 2.5 in. apart along the specimen, and that the impressions made by the standardized testing machine be spaced alternately along the length of the specimen with the impressions made by the machine under calibration.

14. If any determination of hardness, either for the machine under calibration or for the standard machine differs by more than 2 per cent from the mean of the four determinations made with that machine it is recommended that such determination be discarded. If two determinations differ more than 2 per cent from the mean the whole series should be discarded.

15. A Brinell hardness testing machine is acceptable for use over a loading range within which its load-measuring device is correct within 3 per cent.

16. The micrometer microscope or other device for measuring diameter of impression may be calibrated by using it to measure lengths on a standard steel or glass scale. The eye piece of a micrometer microscope should be adjusted so that throughout the range covered the error of reading does not exceed 0.01 mm.

TENTATIVE METHOD OF VERIFICATION OF TESTING MACHINES BY MEANS OF AN ELASTIC CALIBRATION BAR[1]

Serial Designation: E 4 – 24 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1924

1. In this method the testing machine to be verified is tested by comparing its load indications with the amount of elastic deformation of a calibration bar, the deformation being measured with a delicate strainometer.

2. The calibration bar may be either a bar tested in tension or in compression and it shall be fitted with spherical-seated shackles or with a spherical-seated bearing block to insure as nearly axial loading as is possible. The arrangement shown in Fig. 1 or Fig. 2 is suggested for tension bars and the arrangement shown in Fig. 3 is suggested for compression bars.

3. (*a*) The calibration bar shall be of such size that under the greatest load for which the bar is to be used (called its full load) the stress on any cross-section will not be more than 80 per cent of the elastic limit of the material, as determined in accordance with Method II for the Determination of Elastic Limit as described under the definition of the term "Elastic Limit" of the Tentative Definitions of Terms Relating to Methods of Testing (Serial Designation: E 6 – 25 T) of the American Society for Testing Materials[2] on a test specimen cut from the end of the bar after its final heat treatment.

(*b*) Before the calibration bar is used for verification purposes, it shall be loaded to its full load at least six times, and between successive loadings it shall be heated to a temperature of 100° C. (212° F.) for a period of thirty minutes or shall be allowed to rest for a period of 24 hours. The object of this treatment is to adjust the bar to its bearings and to relieve internal strain.

4. The *loading range* of a calibration bar is the range of indicated loads for which the bar gives results within specified tolerances.

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[1] This Tentative Method, when adopted as standard, will become a part of the present Standard Methods of Verification of Testing Machines (Serial Designation: E 4 – 24), 1924 Book of A.S.T.M. Standards.

Criticisms of this Tentative Method are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing of Metallic Materials, University of Illinois, Urbana, Ill.

[2] See p. 798.

Obviously, the loading range may not include loads either greater than the largest load or less than the smallest load applied in verifying the calibration bar.

5. (*a*) The loading range of a calibration bar shall be determined by verifying its indications by the use of standard weights or by standard weights and proving levers. In verifying the indications of the bar, an initial load of 1 per cent of its full load shall first be applied. All changes of strainometer reading and of applied load shall be

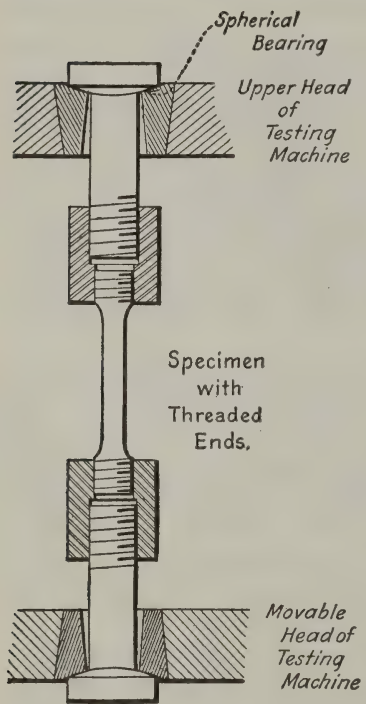


FIG. 1.

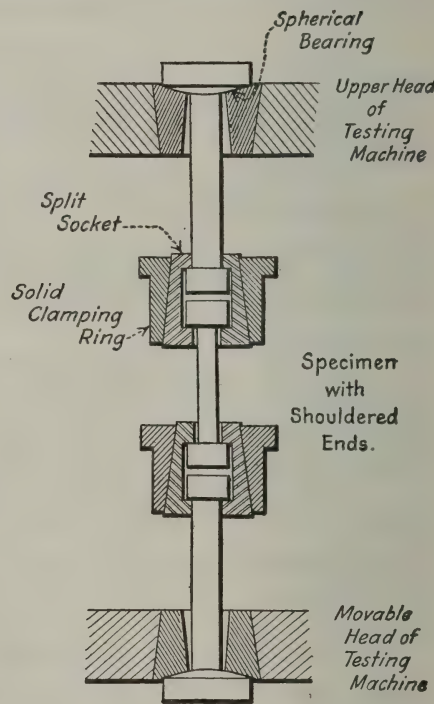


FIG. 2.

reckoned from the readings at this initial load. Successive loads shall be applied, a recommended series of loads being 10, 20, 50 and 100 per cent of the full load of the bar. Such a series shall be applied to the bar at least five times to determine the relation of applied load to strainometer reading, the shackles or bearing blocks being readjusted before each application of the series.

(*b*) The average of the five (or more) strainometer readings obtained in this way shall be taken for each load applied. The average of the differences (taken without regard to algebraic sign) between

the individual strainometer readings for any load and the mean reading for that load constitutes the average deviation for that load. The lowest value of load for which the average deviation is not greater than 0.5 per cent of the strain for that load is the lower limit of the loading range of the calibration bar.

6. The strainometer shall be permanently attached to the calibration bar and shall be so designed as to indicate the average elongation or compression of the bar. The strainometer shall be sufficiently sensitive to indicate a change of 0.25 per cent of the minimum load in the loading range of the bar.

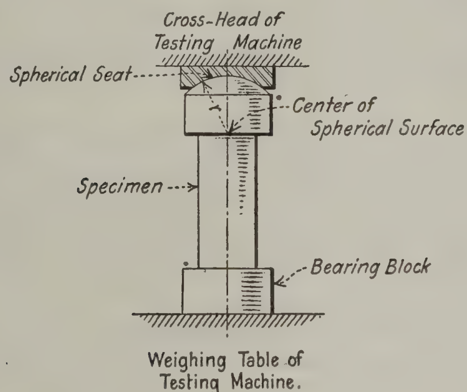


FIG. 3.

7. In using the calibration bar it is important that just previous to taking any reading, including the initial reading of a series, there shall be no reversal in the direction of motion of the indicating mechanism of the strainometer, because such reversal may introduce error due to backlash in the instrument. That is, when taking readings for a series of increasing loads the load should be brought up to each predetermined test load, and when taking readings for a series of decreasing loads the load should be brought down to each predetermined test load.

8. The calibration bar shall be used at a temperature between 10 and 32° C. (50 and 90° F.).

TENTATIVE DEFINITIONS OF TERMS RELATING TO METHODS OF TESTING[1]

Serial Designation: E 6 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to revision.

ISSUED, 1923; REVISED, 1924, 1925.

Stress.—The intensity (measured per unit area) of the internal distributed forces or components of force which resist a change in the form of a body. Stress is measured in force per unit area (pounds per square inch, kilograms per square millimeter, etc.).

NOTE.—In examining the definitions given in various authoritative text-books two definitions of the term "stress" were found. Text-books in physics and some European text-books in mechanics of materials gave a definition substantially as above. American text-books in engineering mechanics define stress as a *force*, measured in *pounds*. They call the intensity of force (pounds per square inch) "unit stress," "intensity of stress," or "fiber stress." This use of the term stress is illustrated by the "stress sheet" of the bridge engineer which gives *forces*, measured in pounds.

In view of the fact that even in engineering text-books the term "stress" is often used to denote intensity of force per unit of area, and in view of the fact that the physicists' definitions involve somewhat simpler terms, the physicists' definitions have been followed in these standards.

While it is important to have a clear definition for the term "stress" it is even more important to keep clearly in mind the *units* used in computations and test data for materials of construction. In fact, if the units are always given, there can be no misunderstanding as to the sense in which the term "stress" is used.

There are three kinds of stress: tensile, compressive, and shearing. Flexure involves the combination of tensile stress and compressive stress. Torsion involves shearing stress.

It is customary to compute stress on the basis of the original dimensions of the cross-section of the body.

Strain.—The change per unit of length in a linear dimension of a body, which change accompanies a stress. Strain is measured in inches per inch of length (millimeters per millimeter).

NOTE.—In American engineering text-books the term "strain" is used in the sense of total deformation and is measured in inches. Change of dimen-

[1] Criticisms of these Tentative Definitions are solicited and should be directed to Mr. H. F. Moore, Chairman of Sub-Committee, of Committee E-1, on Mechanical Testing of Metallic Materials, University of Illinois, Urbana, Ill.

These definitions are in effect a revision of the definitions of terms appearing in the Standard Methods of Mechanical Testing of Metallic Materials. The standard methods, which were last published under the Serial Designation: E 1 – 18, have accordingly been withdrawn.

sion per unit length is called "unit-strain," or "unit deformation." As in the consideration of the term "stress," the definitions given in text-books in physics have been followed.

Under tensile stress or compressive stress, strain is measured along the dimension under consideration. Shearing strain is measured at right angles to the dimension under consideration. In torsion tests, which involve shearing stress, it is customary to measure the angle of twist, which may be translated into terms of strain.

Stress-Strain Diagram.—A diagram plotted with values of stress as ordinates and values of strain as abscissas.

NOTE.—The use of the term stress-strain diagram is frequently extended to cover diagrams plotted with values of applied load, or applied moment as ordinates, and with values of stretch, compression, deflection, or twist as abscissas.

Fig. 1 is an example of a stress-strain diagram.

Stress-strain diagrams are in some cases drawn directly by an autographic attachment to the testing machine. A more usual method of procedure consists in taking a series of load readings (from the balanced scale beam of the testing machine) with corresponding readings of the strain-indicating apparatus (extensometer, compressometer, deflectometer, or torsion indicator). The term "strainometer reading" will be used to denote the reading of any strain-measuring instrument. From these readings, or from values computed from them, there is plotted a diagram with stress-indicating values (load, flexural moment, twisting moment, or stress) as ordinates, and strain-indicating values (elongation, shortening, deflection, twist, or strain) as abscissas. In planning such a test it is necessary to decide on the increment of load or the increment of reading of strainometer to be used between successive readings.

In Fig. 2 are shown three typical stress-strain diagrams. The material for all three tests is the same, and the test specimens are all of the same size, so that the diagrams should be the same. The diagram shown in Fig. 2 (*a*) is plotted from points determined by taking increments of load (S). In this diagram it is seen that owing to the shape of the curve, data for locating points between M and N are lacking. That particular portion of the curve (the "knee" of the curve) is the part for which it is especially desirable to locate several points on the diagram.

In Fig. 2 (*b*) is shown a diagram plotted from points determined by taking increments of strainometer reading (e). It will be noted that for this diagram there are located several points near the knee of the curve, and the shape of the diagram in this important region is much more definitely determined than for the curve shown in Fig. 2 (*a*).

The custom of choosing increments of load rather than increments of strainometer reading is quite common in tests of materials and is followed because, in general, it is easier to compute increments of load than it is to compute increments of strainometer reading. An estimate of the load necessary to stress the specimen up to the knee of the curve is made and some fraction (frequently one-tenth) of this value is taken as an increment. The computation of the corresponding increment of strainometer reading is more complicated.

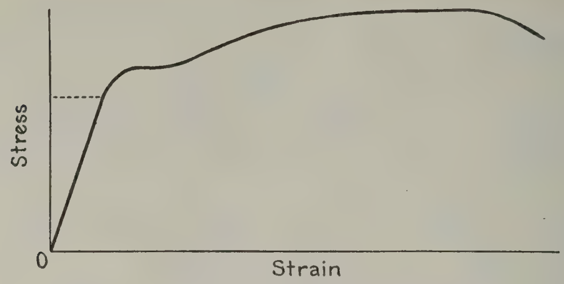
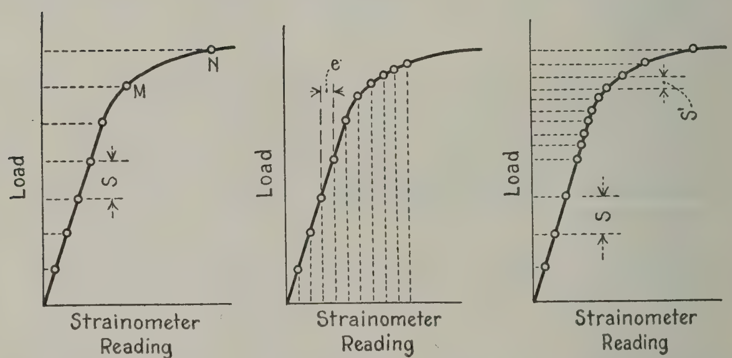


FIG. 1.—A Typical Stress-Strain Diagram.



(*a*) With Equal Increments of Stress.

(*b*) With Equal Increments of Strain.

(*c*) With Two Values of Increments of Stress.

FIG. 2.—Illustrating Three Methods of Plotting Load-Deformation Curves.

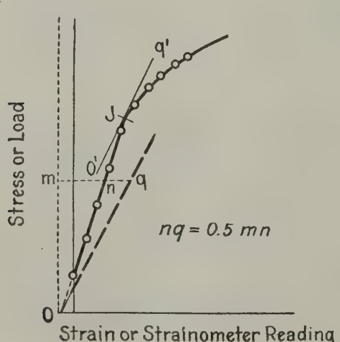


FIG. 3.—Illustrating Method II for Determining Elastic Limit.

Sometimes the practice is followed of applying a few increments of load as determined above, and then applying load in much smaller increments (S') until the knee of the curve is passed. Fig. 2 (c) shows a diagram obtained in this manner. This method involves a marked increase in the number of readings necessary for a test and with unknown material there is always some danger that the knee of the curve will be reached before the use of small increments of load is begun.

The following method of choosing increments for a test is suggested: Estimate the load corresponding to the knee of the stress-strain diagram and choose a value for increment of load about one-tenth of this value. Apply this increment of load (S) once and note the corresponding change of reading for the strainometer (e). Then for the remainder of the test, use for the increment of strainometer reading a value which corresponds to some convenient interval on the scale of the strainometer, and which is approximately equal to (e).

Elastic Limit.—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

NOTE.—In certain specifications of the Society the term "elastic limit" is used to designate a value obtained by a test method which does not involve the release of stress during a test, but which does involve the determination of a limiting load at which there occurs an appreciable change in rate of strain with respect to stress. This limit is frequently given the name "proportional limit" (or "proportional elastic limit"), and the following definition may be given:

Proportional Limit.—The greatest stress which a material is capable of developing without a deviation from the law of proportionality of stress to strain (Hooke's Law).

It is a matter of experience with many materials, especially with many metallic materials, that using ordinary methods of testing, the values found for elastic limit by means of observations of permanent deformation (set) after release of stress do not differ widely from the values found for proportional limit. Since the determination of proportional limit is much more readily made than is the determination of elastic limit it is customary to accept the proportional limit as equivalent to the elastic limit for such materials.

Using any known method, the value determined either for proportional limit or for elastic limit is somewhat arbitrary; the value depends on various test conditions, among them precision of apparatus, the scales to which stress-strain diagrams may be plotted, and the tolerance allowed before it is judged that there exists an appreciable deviation from the law of proportionality of stress to strain or an appreciable permanent deformation (set) upon release of stress.

In connection with this note on elastic limit and proportional limit, it seems proper to consider the action of a material under steadily increasing stress, and to discuss briefly five stages which may be distinguished between the first application of stress and fracture of the metal. Not all materials show all five stages, and for a material the end of one stage and the beginning of the next stage are not always marked.

In the first stage (OA, Fig. 4), with the use of measuring instruments of ordinary sensitiveness, deviations from the law of proportionality of stress to

strain (Hooke's Law) will not be detected, nor will there be detected set after release of stress.

In the second stage (*AB*, Fig. 4) as stress increases there is detected an appreciable and growing deviation from the law of proportionality of stress to strain. This is usually accompanied by an appreciable and growing permanent

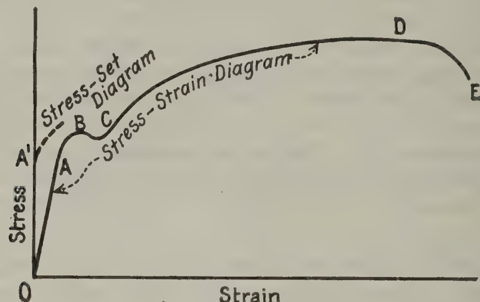


FIG. 4.

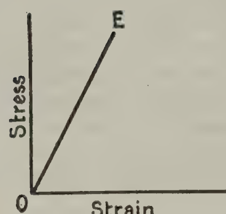


FIG. 5.

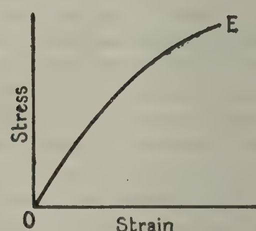


FIG. 6.

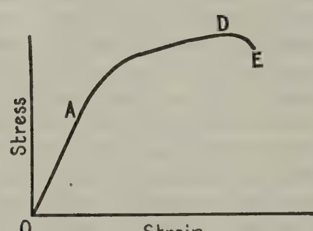


FIG. 7.

deformation (set) after release of stress, although for some materials (*e.g.*, rubber) measurable permanent deformation may not occur after release of stress exceeding the proportional limit. Values for elastic limit and for proportional limit are found in this second stage.

The third stage (*BC*, Fig. 4) is characteristic of ductile metals. In this stage, strain increases without any increase of stress, and in some cases increase of strain is accompanied by an actual decrease of stress. The yield point is found at the beginning of this stage (at *B*, Fig. 4).

In the fourth stage (*CD*, Fig. 4) stress and strain both increase up to the maximum stress carried (*D*, Fig. 4, which gives the tensile strength in a tension test of a material).

In the fifth stage (*DE*, Fig. 4) strain increases while stress diminishes from the maximum value (*D*, Fig. 4) to fracture (*E*, Fig. 4). In considering each of these stages it is important to note that stress is computed on the basis of original cross-section of specimen.

Many materials, especially brittle materials, do not exhibit all the above-mentioned stages. Some (*e.g.*, glass and fused quartz) show only the first stage. Fig. 5 gives a representative stress-stain diagram for such materials. Some materials (*e.g.*, cast iron) show only the second stage, and Fig. 6 shows a typical stress-strain diagram for such materials. Some materials (*e.g.*, medium-hard steel) do not show the third stage, and the second stage merges into the fourth stage. Fig. 7 shows a typical stress-strain diagram for such materials. The fifth stage is absent in many materials; it is, in fact, characteristic of ductile metals only.

The following methods of test are, strictly speaking, methods of determining proportional limit with various degrees of precision. However, the values determined by these methods may, for certain materials, be regarded as satisfactory and reliable values for the elastic limit, and they are therefore so used in certain specifications of the Society.

Method I. (For tension tests only, proposed by Committee A-1 on Steel, and embodied in a number of specifications for steel.) The elastic limit shall be determined by an extensometer reading to 0.0002 in. The extensometer shall be attached to the specimen at the gage marks and not to the shoulders of the specimen nor to any part of the testing machine. When the specimen is in place and the extensometer attached, the testing machine shall be operated so as to increase the load on the specimen at a uniform rate. The observer shall watch the elongation of the specimen as shown by the extensometer and shall note, for this determination, the load at which the rate of elongation shows a sudden increase. The extensometer may then be removed from the specimen and the test continued to determine the tensile strength.

Method II (proposed by the late J. B. Johnson) is a somewhat more delicate method for locating the elastic limit than is Method I. In Method II, the elastic limit shall be taken as that stress at which the rate of deformation is 50 per cent greater than the initial rate of deformation. A stress-strain diagram is necessary for determining the elastic limit by Method II. Fig. 3 illustrates the use of Method II. The initial rate of deformation is given by the ratio $mn: Om$. $nq = 0.5\ mn$, $mq = 1.5\ mn$, and the slope of Oq represents a rate of deformation 50 per cent greater than the initial rate. $O'q'$ is drawn parallel to Oq and tangent to the stress-strain diagram. The point of tangency J locates the elastic limit. In using Method II the same precautions should be observed in attaching the strainometer as for the determination of the elastic limit by Method I. Method II, if used for tension tests of metals, gives lower values for the elastic limit than does Method I. It is recommended that in using Method II for tension tests the strainometer be of such sensitiveness that it will indicate a change of strain of 0.0001 in. per inch of gage length (0.0001 mm. per millimeter of gage length) and that a corresponding degree of sensitiveness be required for strainometers used for compression tests, for flexure tests, and for torsion tests.

When it is desired to determine the elastic limit with a high degree of precision, it is suggested that the experimenter devise his own procedure, and in reporting his results describe the procedure in detail, including a statement of the limits of sensitiveness of the apparatus used and of the procedure used in plotting the stress-strain diagram from which the determination of elastic limit is made. It is to be remembered that the accurate determination of the elastic limit requires the use of accurate and sensitive instruments and the use of accurate methods of plotting test data.

Yield Point.—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

Note.—Some metals do not show a clearly defined yield point.

Two methods are in use for determining the yield point: (I) The "drop of the beam" method, and (II) the method by use of dividers. In Method I, load is applied to the specimen at a steady rate of increase and the operator keeps the beam in balance by running out the poise at a steady rate. At the yield point the increase of load stops, but the operator, running out the poise at a steady rate, runs it a trifle beyond the balance position, and the beam of the machine drops for a brief but appreciable interval of time. In a machine fitted with a self-indicating load-measuring device there is a sudden halt of the load-indicating pointer, corresponding to the drop of the beam. The load at the "halt" or the "drop" is recorded, and the corresponding stress is taken as the yield point. This method of determining the yield point requires only one man to conduct a test.

In Method II, an observer with a pair of dividers watches for visible elongation between two section marks on the specimens. When visible stretch is observed, the load at that instant is noted, and the stress corresponding to the load is taken as the yield point. When a strainometer[1] is used in place of dividers a deformation of 0.01 in. over a gage length of 2 in. shall be regarded as indicating the yield point.

Tensile Strength.—The maximum tensile stress which a material is capable of developing.

Note.—In practice, it is considered to be the maximum stress carried by a specimen representing the material in a tension test to rupture, under definite prescribed conditions. Tensile strength is computed from the maximum load carried during a tension test and the original cross-sectional area of the specimen.

Compressive Strength.—The maximum compressive stress which a material is capable of developing.

Note.—In the case of a material which fails in compression by a shattering fracture the compressive strength has a very definite value. In the case of

[1] The determintion of yield point with a strainometer is sometimes made in tests on non-ferrous metals.

materials which do not fail in compression by a shattering fracture the value obtained for compressive strength is an arbitrary value depending upon the degree of distortion which is regarded as indicating complete failure of the metal.

Modulus of Elasticity.—The ratio, within the elastic limit of a material, of stress to corresponding strain.

NOTE.—As there are three kinds of stress, so are there three moduli of elasticity for any material: the modulus in tension, the modulus in compression, and the modulus in shear. The value of the modulus of elasticity in tension is nearly the same, for most metals, as the value of the modulus of elasticity in compression. The value of the modulus of elasticity in shear is smaller than the value of the modulus of elasticity in tension. The modulus of elasticity is expressed in pounds per square inch (kilograms per square millimeter).

TENTATIVE DEFINITIONS OF TERMS RELATING TO SPECIFIC GRAVITY[1]

Serial Designation: E 12 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

Issued, 1925

1. *Absolute Specific Gravity* (of solids and liquids).—The ratio of the weight referred to vacuum of a given volume of the material at a stated temperature to the weight referred to vacuum of an equal volume of gas-free distilled water[2] at a stated temperature. It shall be stated thus:

(*a*) When the temperature of the material and that of the water are the *same:*

Absolute Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperature of the material and that of the water are *not the same:*

Absolute Specific Gravity $\frac{x° \text{ C.,}}{y° \text{ C.}}$ ....,

where x is the temperature of the material and y the temperature of the water.

2. *Specific Gravity* (of solids and liquids).—The ratio of the weight in air of a given volume of the material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperature of the material xnd that of the water are the *same:*

Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperature of the material and that of the water are *not the same:*

Specific Gravity $\frac{x° \text{ C.,}}{y° \text{ C,}}$ ....,

where x is the temperature of the material and y is the temperature of the water.

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[1] Criticisms of these Tentative Definitions are solicted and should be directed to Mr. F. M. Farmer, Chairman of Sub-Committee, of Committee E-8, on Definition of Specific Gravity, Electrical Testing Laboratories, Eightieth Street and East End Ave., New York City.

[2] Distilled water boiled vigorously in vacuum.

3. *Apparent Specific Gravity* (of solids).—The ratio of the weight in air of a given volume of the impermeable portion of a permeable material (that is, the solid matter including its impermeable pores or voids) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperature of the material and that of the water are the *same:*

Apparent Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperature of the material and that of the water are *not the same:*

Apparent Specific Gravity $\frac{x° \text{ C.,}}{y° \text{ C.}}$ ....,

where x is the temperature of the material and y is the temperature of the water.

NOTE.—In scientific circles, specific gravity and density determinations made in air (that is, uncorrected to vacuum) are frequently distinguished by the adjective "apparent." Thus, the specific gravity defined by definition No. 2 would be designated as "apparent specific gravity" and that defined by definition No. 1 as "specific gravity." But in industry, the terminology is more generally in accordance with that given in these definitions.

4. *Bulk Specific Gravity* (of solids).—The ratio of the weight in air of a given volume of a permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperature of the material and that of the water are the *same:*

Bulk Specific Gravity $x°$ C., ...,

where x is the temperature of the material and the water.

(*b*) When the temperature of the material and that of the water are *not the same:*

Bulk Specific Gravity $\frac{x° \text{ C.,}}{y° \text{ C.}}$ ....,

where x is the temperature of the material and y is the temperature of the water.

NOTES.—The terms "permeable" and "impermeable" cannot be rigidly defined for general application. The exact meaning in a particular application is the conventional one inferred by the procedure specified for determining the specific gravity of the material in question.

It is obvious that in the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed whenever possible.

TENTATIVE DEFINITION OF THE TERM SCREEN (SIEVE)[1]

Serial Designation: E 13 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925

Screen (*Sieve*).—A plate or sheet or a woven cloth, with regularly spaced apertures of uniform size, mounted on a suitable frame or holder, for use in separating materials according to size.

NOTE.—The shape and spacing of apertures, size of wires or threads, thickness of plate or sheet, allowable variations and similar properties, should be taken care of in specifications.

[1] Criticisms of this Tentative Definition are solicited and should be directed to Mr. L. R. Ferguson, Chairman of the Sub-Committee, of Committee E-8, on Definitions of Sieve and Screen, 342 Madison Ave., New York City.

TENTATIVE RECOMMENDED PRACTICE
FOR
THERMAL ANALYSIS OF STEEL[1]

Serial Designation: E 14 – 25 T

This is a **Tentative Standard** only, published for the purpose of eliciting criticism and suggestions. It is not a Standard of the Society and is subject to annual revision.

ISSUED, 1925.

I. APPARATUS

Apparatus. 1. The apparatus required for thermal analysis consists of a furnace for heating the specimen, a thermocouple, a potentiometer or galvanometer for measuring small changes in e. m. f. and a stop-watch or chronometer.

Furnace. 2. An electric-resistance furnace provided with a means of accurate and smooth temperature control should be used. A furnace of the Rosenhain type or U. S. Bureau of Standards modification, is recommended. A good furnace is essential for accurate results. A neutral or reducing atmosphere or partial vacuum is desirable to protect the specimen from excessive oxidation.

Thermocouple. 3. The thermocouple may be of any approved type which has been calibrated either by the Bureau of Standards or against a secondary standard or by the melting point of pure metals (Tin 231.9° C., Lead 327.5° C., Zinc 419.4° C., Aluminum 658.7° C., Silver 960.5° C., Copper 1083° C.). A charge of at least 60 g. of metal should be used.[2] Base metal couples should not be used for temperatures exceeding 900° C.

4. For recording data, a sensitive galvanometer or potentiometer and stop-watch or a recording galvanometer and a chronograph should be used.

II. SPECIMENS

Specimens 5. The size of specimen must be governed by the kind of furnace and the sensitivity of the instruments used. About ½ by ½ by 1 in. is satisfactory for most work.

[1] Criticisms of this Recomended Practice are solicited and should be directed to Mr. G. F. Comstock, Secretary of Committee E-4 on Metallography, 967 Harrison Ave,, Niagara Falls, N. Y.

[2] The couple should be inserted in the specimen, insulated from it with mica or asbestos.

The specimen should be normalized before making a thermal analysis.

Note—This requirements applies to routine work only. For special researches exception may be made.

III. PROCEDURE

Rate of Heating and Cooling. 6. The specimen should be heated and cooled at a uniform rate of from 5 to 10° C. (9 to 18° F.) per minute.

Maximum Temperature. 7. The specimen should be heated to a temperature of 100° C. above the upper critical point and allowed to remain there fifteen minutes before cooling is begun.

Interpretation of Curves. 8. Data may be plotted as desired. The inverse rate method is the simplest and most satisfactory for all general purposes and the most generally used.

In fixing the critical points, the temperature should be read at the cusp, or maximum point, of the inflection of the curve.

TENTATIVE REVISIONS

OF

A.S.T.M. STANDARDS

Tentative revisions of A.S.T.M. Standards are printed for one or more years with a view of eliciting criticisms, of which the committee concerned will take due cognizance before recommending final action.

Members of the Society and others are invited to direct written criticisms of any of these Tentative Revisions to the officer of the appropriate committee, whose name and address appear in the footnote in connection with each revision.

TENTATIVE REVISIONS OF A.S.T.M. STANDARDS

A. FERROUS METALS

STANDARD SPECIFICATIONS FOR CARBON-STEEL AND ALLOY-STEEL FORGINGS

(SERIAL DESIGNATION: A 18 – 21)[1]

STANDARD SPECIFICATIONS FOR QUENCHED-AND-TEMPERED CARBON-STEEL AXLES, SHAFTS, AND OTHER FORGINGS FOR LOCOMOTIVES AND CARS

(SERIAL DESIGNATION: A 19 – 21)[1]

STANDARD SPECIFICATIONS FOR CARBON-STEEL FORGINGS FOR LOCOMOTIVES

(SERIAL DESIGNATION: A 20 – 21)[1]

STANDARD SPECIFICATIONS FOR QUENCHED-AND-TEMPERED ALLOY-STEEL AXLES, SHAFTS, AND OTHER FORGINGS FOR LOCOMOTIVES AND CARS

(SERIAL DESIGNATION: A 63 – 21)[1]

In Specifications A 18 – 21 change Section 4, and in Specifications A 20 – 21 change Section 5 from their present form: namely,

"The manufacturer and the purchaser shall agree upon forgings on which a prolongation for test purposes shall be provided."

and in Specifications A 19 – 21 and A 63 – 21 change Section 4 from its present form: namely,

"For test purposes, a prolongation shall be left on each forging, unless otherwise specified by the purchaser."

to read as follows:

"Unless otherwise specified, for test purposes at least 20 per cent of the forgings shall be provided with prolongations or, at the manufacturer's option, a forging may be selected."

In Specifications A 18 – 21 and A 20 – 21 change Section 17 (*a*), and in Specifications A 19 – 21 and A 63 – 21 change Section 19 (*a*) from their present form: namely,

"The inspector representing the purchaser shall have free entry at all times while work on the contract of the purchaser is being performed, to all

[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

parts of the manufacturer's works which concern the manufacture of the forgings ordered. The manufacturer shall afford the inspector, free of cost, all reasonable facilities to satisfy him that the forgings are being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment."

to read as follows:

"The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the forgings ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the forgings are being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works."

STANDARD SPECIFICATIONS FOR CARBON-STEEL CAR AND TENDER AXLES

(SERIAL DESIGNATION: A 21 – 18)[1]

A revision in the form of separate tentative specifications entitled "Specifications for Carbon-Steel Car and Tender Axles (A 21 – 25 T)"[2] is intended to replace the present standard specifications.

STANDARD SPECIFICATIONS FOR WELDED AND SEAMLESS STEEL PIPE

(SERIAL DESIGNATION: (A 53 – 24)[1]

Section 1.—Change from its present form: namely,

"These specifications cover 'standard,' 'extra strong,' and 'double extra strong' welded and seamless steel pipe. Pipe ordered under these specifications is intended for bending, flanging and other special purposes."

to read as follows:

"*Scope.*—These specifications cover 'standard weight,' 'extra strong,' and 'double extra strong' welded and seamless steel pipe. Pipe ordered under these specifications is intended for coiling, bending, flanging and other special purposes. Butt-welded pipe is not intended for flanging."

STANDARD SPECIFICATIONS FOR LAP-WELDED AND SEAMLESS STEEL AND LAP-WELDED IRON BOILER TUBES

(SERIAL DESIGNATION: A 83 – 24)[1]

Section 16.—Insert a new Paragraph (*e*) to read as follows, relettering the subsequent paragraph accordingly:

"(*e*) The gaging tolerances in Paragraphs (*b*), (*c*) and (*d*) apply only to tubes as rolled or drawn and before swaging and expanding."

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. G. H. Woodroffe, Secretary of Committee A-1 on Steel, Reading Iron Co., Reading, Pa.

[2] See p. 53.

STANDARD SPECIFICATIONS FOR WELDED WROUGHT-IRON PIPE

(SERIAL DESIGNATION: A 72 – 24)[1]

Section 1.—Change from its present form: namely,

"These specifications cover 'standard,' 'extra strong' and 'double extra strong' welded wrought-iron pipe."

to read as follows:

"These specifications cover 'standard weight,' 'extra strong' and 'double extra strong' welded wrought-iron pipe. Pipe ordered to these specifications is intended for coiling, bending, flanging, and other special purposes. Butt-welded pipe is not intended for flanging."

Section 3 (b).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"(*b*) All pipe [3 in. or under] *under 2 in.* in nominal diameter may be butt-welded, unless otherwise specified. All pipe [over 3 in.] *2 in. or over* in nominal diameter shall be lap welded."

Section 9.—Add a new Section 9 to follow the present Section 8 to read as follows, renumbering the subsequent sections accordingly:

"In case of doubt as to the presence of steel, the purchaser may, at his own expense, make micrographic examination and chemical analysis to assist in determining whether the material meets the requirements of Section 3 (*a*)."

STANDARD SPECIFICATIONS FOR STAYBOLT, ENGINE-BOLT AND EXTRA-REFINED WROUGHT-IRON BARS

(SERIAL DESIGNETION: A 84 – 24)[1]

Section 7.—Add two new Paragraphs (*b*) and (*c*) to read as follows, re-lettering the present Paragraph (*b*) to Paragraph (*d*):

"(*b*) *Grade A.*—For bars $1\frac{1}{4}$ sq. in. in sectional area, the following deductions from the minimum requirements specified in Section 6 (*a*) shall be made for each square inch of nominal section above $1\frac{1}{4}$ sq. in.:

Tensile strength, 250 lb., but not under 46,000 lb.

Reduction of area, 3 per cent, but not under 40 per cent.

"(*c*) *Grade A.*—For flats of all sizes, the minimum reduction of area shall be 40 per cent."

STANDARD SPECIFICATIONS FOR MALLEABLE CASTINGS

(SERIAL DESIGNATION: A 47 – 24)[2]

Section 3.—Add to the table of requirements a requirement on minimum yield point, as follows:

"Yield point, lb. per sq. in., minimum........ 30 000"

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. C. C. Osterhout, Secretary of Committee A-2 on Wrought Iron, Rome Iron Mills, Inc., Rome, N. Y.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. Enrique Touceda, Secretary of Committee A-7 on Malleable Castings, 943 Broadway, Albany, N. Y.

Add a new paragraph (*b*) to read as follows:

"(*b*) The yield point shall be determined by the drop of the beam of the testing machine."

RECOMMENDED PRACTICE FOR HEAT TREATMENT OF CASE-HARDENED CARBON-STEEL OBJECTS

(SERIAL DESIGNATION: A 37 – 14)[1]

A revision in the form of a separate tentative recommended practice entitled "Recommended Practice for Carburizing and Heat Treatment of Carburized Objects (A 37 – 25 T)"[2] is intended to replace the present recommended practice.

B. NON-FERROUS METALS

STANDARD SPECIFICATIONS FOR LAKE COPPER WIRE BARS, CAKES, SLABS, BILLETS, INGOTS, AND INGOT BARS

(SERIAL DESIGNATION: B 4 – 13)[3]

Section 4 (*a*).—Change the first sentence in the second paragraph to read as follows by the addition of the italicized figures and the omission of those in brackets:

"Low Resistance Lake wire bars shall have a resistivity not to exceed [0.15535] *0.15436* international ohms per meter-gram at 20° C. (annealed)."

Section 5 (*a*).—Change to read as follows by the addition of the italicized figures and the omission of those in brackets:

"Low Resistance Lake copper shall have a purity of at least [99.880] *99.900* per cent as determined by electrolytic assay, silver being counted as copper."

Section 5 (*b*).—Change the first sentence to read as follows by the addition of the italicized figures and the omission of those in brackets:

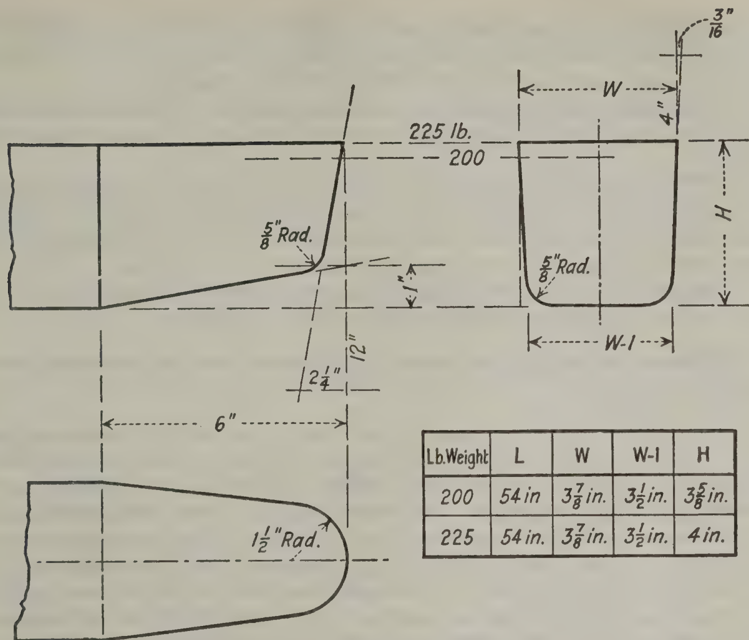
"High Resistance Lake copper shall have a purity of at least [99.880] *99.900* per cent, copper, silver, and arsenic being counted together."

Section 7.—Add a new Section 7 to read as follows, renumbering the subsequent sections accordingly:

[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. J. H. Hall, Secretary of Committee A-4 on Heat Treatment of Iron and Steel, Taylor-Wharton Iron and Steel Co., High Bridge, N. J.

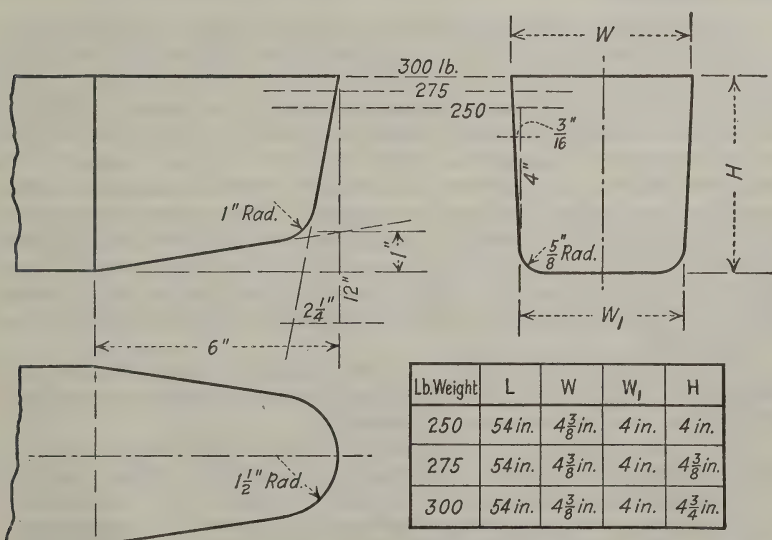
[2] See p. 116.

[3] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.



Lb.Weight	L	W	W-1	H
200	54 in	$3\frac{7}{8}$ in.	$3\frac{1}{2}$ in.	$3\frac{5}{8}$ in.
225	54 in.	$3\frac{7}{8}$ in.	$3\frac{1}{2}$ in.	4 in.

FIG. 1.



Lb.Weight	L	W	W_1	H
250	54 in.	$4\frac{3}{8}$ in.	4 in.	4 in.
275	54 in.	$4\frac{3}{8}$ in.	4 in.	$4\frac{3}{8}$ in.
300	54 in.	$4\frac{3}{8}$ in.	4 in.	$4\frac{3}{4}$ in.

FIG. 2.

"7. *Standard Sizes and Shapes for 200 to 300-lb. Wire Bars.*—(*a*) One mold shall be used for casting 200 to 230-lb. wire bars, the bottom width of this bar to be $3\frac{1}{2}$ in., the listed weights being 200 and 225 lb. (See Fig. 1.)

"(*b*) One mold shall be used for casting 240 to 300-lb. wire bars, the bottom width of this bar to be 4 in., the listed weights being 250, 275 and 300 lb. (See Fig. 2.)

"(*c*) The length dimension of all bars shall be 54 in. The side draft or taper shall be $\frac{3}{8}$ in. in 4 in. ($\frac{3}{16}$ in. in 4 in. on each side of the bar). The radius of the corners at the bottom of the bars shall be $\frac{5}{8}$ in. The end taper at the bottom shall be 6 in. long overall and approximately 2 in. per ft.

"The end taper of the side shall be approximately $2\frac{1}{4}$ in. per ft. and the end of the bar shall be approximately $3\frac{3}{8}$ in. deep at the point."

Section 9.—Change the second paragraph from its present form: namely,

"In a question of metal contents each party shall select a sample of two pieces. These shall be drilled in the presence of both parties, several holes approximately $\frac{1}{2}$ in. in diameter being drilled completely through each piece; scale from set shall be rejected. No lubricant shall be used and drilling shall not be forced sufficiently to cause oxidation of chips. The resulting samples shall be cut up, mixed, and separated into three parts, each of which shall be placed in a sealed package, one for each party and one for the umpire if necessary. Each party shall make an analysis, and if the results do not establish or dismiss the claim to the satisfaction of both parties the third sample shall be submitted to a mutually agreeable umpire, who shall determine the question of fact, and whose determination shall be final."

to read as follows:

"In a question of metal contents, each party shall select a sample of three pieces from the consignment or lot to be investigated. These shall be sampled in the presence of both parties by drilling five holes, approximately $\frac{1}{2}$ in. in diameter, at points equally spaced between the ends of the pieces. With bars, billets, ingots, and ingot bars, these holes should be along a middle line and with slabs and cakes on a diagonal line projected between opposite corners. The drilling shall be from top to bottom and completely through each piece. Scale from set and any surface dirt shall be rejected. No lubricant shall be used and drilling shall not be forced sufficiently to cause oxidation of the chips. In the case of sections having a depth greater than 5 in., drillings may be made from both top and bottom for a depth of not less than 2 in. in each direction instead of completely through, but the drilling shall be otherwise conducted as before described.

"The resulting sample shall be cut up, mixed and separated into three equal portions, each of which shall be placed in a sealed package, one for each party and the third for the umpire, if necessary.

"Each party shall make an analysis by the Standard Method of Battery Assay of Copper (B 34 - 20) of the American Society for Testing Materials and if the results do not establish or dismiss the claim to the satisfaction of both parties concerned, the third sample shall be submitted to a mutually agreeable umpire, who shall determine the question of fact and whose determination shall be final."

STANDARD SPECIFICATIONS FOR ELECTROLYTIC COPPER WIRE BARS, CAKES, SLABS, BILLETS, INGOTS, AND INGOT BARS

(SERIAL DESIGNATION: B 5 – 13)[1]

Sections 3 (a).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"*Metal Content.*—The copper in all shapes shall have a purity of at least [99.880] *99.900* per cent, as determined by [electrolytic assay] *the A.S.T.M. Standard Method of Battery Assay of Copper (B 34 – 20)*, silver being counted as copper."

Section 3 (b).—Change to read as follows by the addition of the italicized figures and the omission of those in brackets:

"*Resistivity.*—All wire bars shall have a resistivity not to exceed [0.15535] *0.15436* international ohms per meter-gram at 20° C. (annealed); all ingots and ingot bars shall have a resistivity not to exceed 0.15694 international ohms per meter-gram at 20° C. (annealed)."

Section 6.—Add a new Section 6 to agree with the new Section 7 added to Specifications B 4 – 13 above, renumbering the subsequent sections accordingly.

Section 7.—Change the second paragraph from its present form: namely,

"In a question of metal contents each party shall select a sample of two pieces. These shall be drilled in the presence of both parties, several holes approximately ½ in. in diameter being drilled completely through each piece; scale from set shall be rejected. No lubricant shall be used and drilling shall not be forced sufficiently to cause oxidation of chips. The resulting samples shall be cut up, mixed, and separated into three parts, each of which shall be placed in a sealed package, one for each party and one for the umpire if necessary. Each party shall make an analysis, and if the results do not establish or dismiss the claim to the satisfaction of both parties the third sample shall be submitted to a mutually agreeable umpire, who shall determine the question of fact, and whose determination shall be final."

to read as follows:

"In a question of metal contents, each party shall select a sample of three pieces from the consignment or lot to be investigated. These shall be sampled in the presence of both parties by drilling five holes approximately ½ in. in diameter, at points equally spaced between the ends of the pieces. With bars, billets, ingots, and ingot bars, these holes should be along a middle line and with slabs and cakes on a diagonal line projected between opposite corners. The drilling shall be from top to bottom and completely through each piece. Scale from set and any surface dirt shall be rejected. No lubricant shall be used and drilling shall not be forced sufficiently to cause oxidation of the chips. In the case of sections having a depth greater than 5 in., drillings may be made from both top and bottom for a depth of not less than 2 in. in each direction instead

[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

of completely through, but the drilling shall be otherwise conducted as before described.

"The resulting sample shall be cut up, mixed and separated into three equal portions, each of which shall be placed in a sealed package, one for each party and the third for the umpire, if necessary.

"Each party shall make an analysis and if the results do not establish or dismiss the claim to the satisfaction of both parties concerned, the third sample shall be submitted to a mutually agreeable umpire, who shall determine the question of fact and whose determination shall be final."

STANDARD SPECIFICATIONS FOR LIGHT ALUMINUM CASTING ALLOYS

(SERIAL DESIGNATION: B 26 – 21)[1]

A revision in the form of separate tentative specifications entitled "Specifications for Aluminum Base Alloy Sand Castings (B 26–25 T)"[2] is intended to replace the present standard specifications.

STANDARD SPECIFICATIONS FOR HIGH-STRENGTH BRONZE TROLLEY WIRE, ROUND AND GROOVED: 40 AND 65 PER CENT CONDUCTIVITY

(SERIAL DESIGNATION: B 9 – 16)[3]

A revision in the form of separate tentative specifications entitled "Specifications for Bronze Trolley Wire (B 9 – 25 T)"[4] is intended to replace the present standard specifications.

STANDARD SPECIFICATIONS FOR SEAMLESS ADMIRALTY CONDENSER TUBES AND FERRULE STOCK

(SERIAL DESIGNATION: B 44 – 24)[1]

Section 12.—Insert under Sub-Heading IV on Permissible Variations in Dimensions, a new Section 12 to read as follows, renumbering the subsequent sections accordingly:

"12. *Diameter.*—The outside diameter of the tubes shall not vary from that specified by more than the following amounts, as measured by 'go' and 'no go' ring gages:

NOMINAL DIAMETER, IN.	PERMISSIBLE VARIATION, IN.
Up to 0.500, inclusive	plus or minus 0.002
Over 0.500 to 0.740, inclusive	" " " 0.0025
Over 0.740 to 1.000, "	" " " 0.003
Over 1.000 to 1.250, "	" " " 0.0035
Over 1.250 to 1.500, "	" " " 0.004

[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

[2] See p. 128.

[3] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. J. A. Capp, Chairman of Committee B-1 on Copper Wire, General Electric Co., Schenectady, N. Y.

[4] See p. 132.

STANDARD SPECIFICATIONS FOR HIGH SHEET BRASS

(SERIAL DESIGNATION: B 36 – 21)[1]

Table I.—Change Table I in Section 13, covering permissible variations in thickness, to agree with the accompanying Table I.

TABLE I.—PERMISSIBLE VARIATIONS IN THICKNESS OF HIGH SHEET BRASS, IN.

Thickness		Width, in.			
B. & S. Gage No.	Inch	Up to 6 in., inclusive	Over 6 to 9 in., inclusive	Over 9 to 14 in., inclusive	Over 14 to 20 in., inclusive
0000	0.460	0.0043	0.0045	0.0047	0.0049
000	0.4096	0.0042	0.0044	0.0046	0.0048
00	0.3648	0.0041	0.0043	0.0045	0.0047
0	0.3249	0.0040	0.0042	0.0044	0.0046
1	0.2893	0.0039	0.0041	0.0043	0.0045
2	0.2576	0.0038	0.0040	0.0042	0.0044
3	0.2294	0.0037	0.0039	0.0041	0.0043
4	0.2043	0.0036	0.0038	0.0040	0.0042
5	0.1819	0.0035	0.0037	0.0039	0.0041
6	0.1620	0.0034	0.0036	0.0038	0.0040
7	0.1443	0.0033	0.0035	0.0037	0.0039
8	0.1285	0.0032	0.0034	0.0036	0.0038
9	0.1144	0.0031	0.0033	0.0035	0.0037
10	0.1019	0.0030	0.0032	0.0034	0.0036
11	0.0907	0.0029	0.0031	0.0033	0.0035
12	0.0808	0.0028	0.0030	0.0032	0.0034
13	0.0719	0.0027	0.0029	0.0031	0.0033
14	0.0640	0.0026	0.0028	0.0030	0.0032
15	0.0570	0.0025	0.0027	0.0029	0.0031
16	0.0508	0.0024	0.0026	0.0028	0.0030
17	0.0452	0.0023	0.0025	0.0027	0.0029
18	0.0403	0.0022	0.0024	0.0026	0.0028
19	0.0359	0.0021	0.0023	0.0025	0.0026
20	0.0320	0.0020	0.0021	0.0023	0.0024
21	0.0284	0.0019	0.0020	0.0021	0.0022
22	0.0253	0.0018	0.0019	0.0020	0.0021
23	0.0225	0.0017	0.0018	0.0019	0.0020
24	0.0201	0.0016	0.0017	0.0018	0.0019
25	0.0179	0.0015	0.0016	0.0017	0.0018
26	0.0159	0.0014	0.0015	0.0016	0.0017
27	0.0142	0.0013	0.0014	0.0015	0.0016
28	0.0126	0.0012	0.0013	0.0014	0.0015
29	0.0112	0.0011	0.0012	0.0013	0.0014
30	0.0100	0.0011	0.0012	0.0013	0.0014
31	0.0089	0.0010	0.0011	0.0012	0.0013
32	0.0079	0.0010	0.0011	0.0012	0.0013
33	0.0070	0.0009	0.0010	0.0011	0.0012
34	0.0063	0.0009	0.0010	0.0011	0.0012
35	0.0056	0.0008	0.0009	0.0010	0.0011
36	0.0050	0.0008	0.0009	0.0010	0.0011

STANDARD SPECIFICATIONS FOR NAVAL BRASS RODS FOR STRUCTURAL PURPOSES

(SERIAL DESIGNATION: B 21 – 19)[1]

Section 6.—Add a paragraph to read as follows:

"(*b*) Yield point in tension shall be determined as the stress producing an elongation under load of 0.5 per cent, that is, 0.01 in. in a gage length of 2 in."

[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. P. D. Merica, Secretary of Committee B-2 on Non-Ferrous Metals and Alloys, International Nickel Co., 67 Wall St., New York City.

C. CEMENT, LIME, GYPSUM AND CLAY PRODUCTS

STANDARD SPECIFICATIONS AND TESTS FOR PORTLAND CEMENT

(SERIAL DESIGNATION: C 9 – 21)[1]

A revision in the form of a separate tentative specification entitled "Specifications and Tests for Compressive Strength of Portland Cement Mortars (C 9 – 16 T)"[2] is intended to become a part of the present standard specifications.

STANDARD SPECIFICATIONS FOR CEMENT-CONCRETE SEWER PIPE

(SERIAL DESIGNATION: C 14 – 24)[3]

Section 1.—Add the following footnote:

"*Caution.*—The consumer or purchaser is cautioned against using cement-concrete pipe where the sewage shows an acid reaction."

STANDARD SPECIFICATIONS FOR FIRE TESTS OF MATERIALS AND CONSTRUCTION

(SERIAL DESIGNATION: C 19 – 18)[4]

Section 2 (a).—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

"The temperature fixed by the curve shall be deemed to be the average [true] temperature [of the furnace gases as] obtained from the readings of several thermo-couples (not less than three) symmetrically disposed and distributed [in such a manner as] to show [temperatures of the gases] *the temperature* near all parts of the sample, *said thermo-couples being enclosed in sealed porcelain tubes of $\frac{3}{4}$-in. outside diameter and $\frac{1}{8}$-in. wall thickness, the immersion of the couple in the furnace chamber to be not less than 12 in. Other types of protecting tubes or pyrometers may be used, that under test conditions give the same indications as the above standard within the limit of accuracy that applies for furnace temperature measurements.*

"*For greater difference of design and size the time temperature curve followed shall be modified to give an exposure equal to that obtained by using the standard pyrometer and curve above described.*"

Section 6.—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. B. Young, Secretary of Committee C-1 on Cement, Hydro-Electric Power Commission of Ontario, 8 Strachan Ave., Toronto, Ont., Canada.

[2] See p. 193.

[3] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. E. S. Rankin, Secretary of Committee C-4 on Clay and Cement-Concrete Sewer Pipe, Engineer in Charge, Bureau of Sewers, City Hall, Newark, N. J.

[4] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. P. Miller, Secretary of Committee C-5 on Fireproofing, 342 Madison Ave., New York City.

"The fire test on the sample with its applied load, if any, shall be continued until failure occurs, or until it has withstood the test conditions for a period equal to [$1\frac{1}{4}$ times that for which classification is desired] *that herein specified in the Conditions of Acceptance for the given type of construction.*"

Section 7.—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

"A second test with duplicate sample shall be made to determine the effect of a hose stream on a sample under fire test, the water being applied at the end of a period equal to [three-fourths] *one-half* of that for which classification is desired, but not later than one hour after the beginning of the test; except that for classification periods of one-half hour or less the fire stream test may be omitted."

TABLE II

Parts of Structure	Type of Protection	Size of Hose Nozzle, in.	Water Pressure at Nozzle, lb.	Time of Application, min.
Floors and Roofs	4 hour	1 1/8	50	[10] *7*
	2 "	1 1/8	50	[5] *4*
	1 "	1 1/8	50	[2.5] *2*
	1/2 "	1 1/8	30	1
	1/4 "	1 1/8	[15] *30*	1
Walls, [Columns] and Partitions	4 "	1 1/8	50	5
	2 "	1 1/8	30	3
	1 "	1 1/8	30	[2.5] *2*
	1/2 "	1 1/8	30	1
	1/4 "	1 1/8	[15] *30*	1
Columns	*4* "	*1 1/8*	*50*	*3*
	2 "	*1 1/8*	*30*	*2*
	1 "	*1 1/8*	*30*	*1 1/2*
	1/2 "	*1 1/8*	*30*	*1*
	1/4 "	*1 1/8*	*30*	*1/2*

Table I.—Change Table I, referred to in Section 8, to read as indicated in the accompanying Table II by the addition of the italicized word and figures and the omission of the word and figures in brackets.

Section 9.—Change to read as follows by the addition of the italicized words and the omission of the words in brackets, changing the side heading from "Loading" to "Loading and Additional Tests":

"For any material or construction intended to carry load other than its own weight, the full [rated safe] working load shall be applied during the entire fire test, also during the fire stream test. After completion of the fire stream test, the sample shall be subjected to [excess loading as prescribed under specifications for the different structural parts] *additional tests as herein specified in the Conditions of Acceptance for the given type of construction.*"

Add a new Section 10, under the sub-division "Conduct of Fire Tests," entitled "Time of Testing," to read as follows:

"The material or construction shall not be tested until a large proportion of its final strength has been attained and the excess of free water has been given off. Where this cannot be attained within 40 days, artificial drying with air temperature not exceeding 100° F. shall be used."

Section 10.—Renumber the present Section 10 to Section 12.

Section 11.—Omit this section reading as follows:

"The floor or roof may be tested as soon after construction as desired, but within 40 days. Artificial drying will be allowed if desired."

Section 12.—Transfer this section, placing it under the subdivision "Conduct of Fire Tests," renumbering it Section 11.

Section 14.—Change the side heading to read "Conditions of Acceptance."

Section 14 (a).—Change to read as follows by the omission of the words in brackets:

"The floor or roof shall have sustained safely the full [rated safe] working load during the fire test without passage of flame for a period equal to $1\frac{1}{4}$ times that for which classification is desired."

Section 18.—Change the side heading to read "Conditions of Acceptance."

Add five new sections relating to column tests to read as follows:

19. *Size of Sample.*—For column tests, the length of the column exposed to fire shall approximate the clear length of the member in the structure, and for building columns shall be not less than 9 ft. The outside lateral dimensions shall be the minimum for which classification is desired. The details of construction, and exposed connections and protection, if any, shall be applied according to the methods of acceptable field practice.

20. *Loading.*—During the fire test and the fire and water test, a constant working load shall be applied to the column, calculated according to the governing design regulations. Provisions shall be made for transmitting the load to the exposed portion without unduly increasing the effective column length.

21. *Measurements.*—Measurements shall be made of the expansion or depression at the head of the column and determination of the period of expansion. Such other measurements shall be made of deformation and of temperature of the column as will give the most useful information for the given column type.

22. *Position of Nozzle.*—The distance of the nozzle from the column during the application of water shall be 20 ft., the water being applied in succession, as uniformly as possible, over the full height on three of its sides.

23. *Conditions of Acceptance.*—The test shall not be regarded as successful unless the following conditions are met:

(*a*) The column shall have sustained the full working load during the fire test for a period equal to —[1] times that for which classification is desired.

[1] The quantity to be inserted in this blank space will be supplied by the committee, when the information is available.

(*b*) The column shall have sustained the full working load during the fire stream test as prescribed in Sections 7 and 8, and after its completion and when cold shall sustain a total load equal to the dead load component plus twice the live load component of its design working load.

(*c*) After the fire stream test the column when dry shell withstand, under working load, a fire test of duration equal to —[1] of the period for which classification is desired, but not eEceeding one hour.

STANDARD SPECIFICATIONS FOR PAVING BRICK

(SERIAL DESIGNATION: C 7 – 15)[2]

In the Appendix to the specifications, eliminate one of the recognized sizes and types of paving brick, making the recommendations for sizes read as follows:

	WIDTH, IN.	DEPTH, IN.	LENGTH, IN.
Plain wire-cut brick (vertical fiber, lugless).......	3^a	4^a	$8\frac{1}{2}$
Plain wire-cut brick (vertical fiber, lugless).......	$3\frac{1}{2}{}^a$	4^a	$8\frac{1}{2}$
Repressed lug brick........................	$3\frac{1}{2}$	4	$8\frac{1}{2}$
Wire-cut lug brick (Dunn)....................	$3\frac{1}{2}$	4	$8\frac{1}{2}$

[a] These bricks are customarily laid in the pavement to give a depth of 3 or $3\frac{1}{2}$ in., respectively, by 4 in. in width and $8\frac{1}{2}$ in. in length.

STANDARD SPECIFICATIONS FOR HYDRATED LIME FOR STRUCTURAL PURPOSES

(SERIAL DESIGNATION: C 6 – 24)[3]

Section 12.—Change the second and fifth paragraphs from their present form: namely,

"The lime shall be made into a stiff putty with water and permitted to soak over night. It shall be molded in a rubber ring such as is used with a Vicat needle, resting the specimen on a glass plate."

"If the penetration is less than standard, the sample may be removed from the mold, mixed with more water, and retested. If the penetration is more than standard, the sample shall be discarded and a new one prepared."

to read respectively as follows:

"The lime shall be made into a stiff putty with water, stirred vigorously with a trowel or spatula for three minutes and permitted to soak over night. It shall be stirred vigorously with a trowel or spatula for three minutes, molded in a rubber ring such as is used with a Vicat needle, resting the specimen on a glass plate."

[1] The quantity to be inserted in this blank space will be supplied by the committee, when the information is available.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. W. E. Emley, Secretary of Committee C-3 on Brick, U. S. Bureau of Standards, Washington, D. C.

[3] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. P. Brown, Secretary of Committee C-7 on Lime, National Lime Association, 918 G St., N. W., Washington, D. C.

"If the penetration is less than standard, the sample may be removed from the mold, mixed with more water, stirred vigorously with a trowel or spatula for three minutes, and retested. If the penetration is more than standard, the sample shall be discarded and a new one prepared."

STANDARD METHODS OF TESTING GYPSUM AND GYPSUM PRODUCTS

(SERIAL DESIGNATION: C 26 – 23)[1]

Add the following sections to the present Standard Methods:

XII. COMPRESSIVE STRENGTH OF GYPSUM PARTITION TILE OR BLOCK

23. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight at a temperature not exceeding 110° F. (43° C.), until two successive weighings of the same specimen do not show a variation in excess of 0.5 per cent. At the option of the manufacturer or purchaser, strength tests may be conducted on samples that are dried at room temperature. However, in cases of controversy all test samples shall be dried to constant weight as herein prescribed.

(*c*) The tile or block shall be tested in the position in which they are designed to be used and shall be bedded on and capped with a felt gasket not less than $\frac{1}{8}$ in. nor more than $\frac{1}{4}$ in. in thickness. At the option of the manufacturer or purchaser, or in cases of controversy, the test samples may be suitably bedded and capped with neat gypsum mortar, or the bearing surfaces of the tile may be planed or rubbed smooth and true. When neat gypsum mortar is used for bedding and capping, the test may be conducted after the mortar has set, but not sooner than one hour after the sample has been capped.

(*d*) The loading head shall completely cover the bearing area of the tile, and the applied load shall be transmitted through a spherical bearing block of proper design. The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute.

XIII. TRANSVERSE STRENGTH OF GYPSUM PARTITION TILE OR BLOCK

24. (*a*) Not less than ten full-size gypsum tile specimens shall be used.

(*b*) The test specimens shall be dried to constant weight as prescribed for the determination of the compressive strength.

(*c*) The tile or block shall be supported on its face (flat) on fixed parallel bearings spaced 24 in. (600 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{2}$ in. (12.7 mm.).

(*d*) The speed of the moving head of the testing machine shall not be more than 0.05 in. per minute. The modulus of rupture shall be calculated in pounds per square inch from the formula:

$$\text{Modulus of Rupture} = \frac{3wl}{2bd^2}$$

where l = distance between supports in inches;
b = breadth (width) of tile in inches;
d = thickness of tile in inches;
w = load in pounds at failure of test specimen.

[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. Virgil G. Marani, Secretary of Committee C-11 on Gypsum, 844 Rush St., Chicago, Ill.

XIV. ABSORPTION OF GYPSUM PARTITION TILE OR BLOCK

25. (*a*) The absorption shall be determined upon a test specimen of partition tile or block consisting of approximately one-half of a full-size tile.

(*b*) The test specimen shall be dried to constant weight as prescribed for the determination of the compressive strength.

(*c*) The dried test specimen shall be submerged in water until a constant saturated weight is attained, but in no event for a period of less than two hours, the water temperature being maintained at between 70 and 80° F. (21 and 27° C.). The test specimen shall be removed, the surface wiped off with a damp cloth and the specimen weighed immediately. The percentage of absorption shall be calculated on the dry weight from the formula:

$$\text{Percentage of Absorption} = \frac{100\ (B-A)}{A}$$

where A = weight of dry test specimen;

B = weight of saturated test specimen.

(*d*) The rate of absorption shall be determined by sealing upon the face of the test specimen, which shall first be dried to constant weight as prescribed for determination of the compressive strength, a glass tube $1\frac{1}{2}$ in. in diameter and 12 in. in length, graduated in centimeters and located centrally over a core. Into this tube shall be poured 250 cc. of water at a temperature of between 70 and 80° F. (21 and 27° C.). The rate of absorption shall be reported in cubic centimeters per minute.

XV. TRANSVERSE STRENGTH OF GYPSUM BOARDS

26. (*a*) Test specimens shall be taken from not less than ten gypsum boards.

(*b*) Strength test specimens shall be 12 in. (300 mm.) in width and approximately 18 in. (460 mm.) in length. They shall be supported on fixed parallel bearings spaced 16 in. (410 mm.) on centers, and shall be loaded through a similar bearing midway between the supports. All bearing and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{8}$ in. (3.2 mm.).

(*c*) Test loads shall be applied at a uniform rate of 60 lb. (27.22 kg.) per minute with a permissible variation of ± 10 per cent in the rate. Strength test results shall be reported when the load is applied across the fiber of the surfacing and also parallel to the fiber of the surfacing.

D. MISCELLANEOUS MATERIALS

STANDARD SPECIFICATIONS FOR TURPENTINE

(SERIAL DESIGNATION: D 13 – 24)[1]

Title.—Change to read as follows:

"Specifications for Gum Spirits of Turpentine and for Steam-Distilled Wood Turpentine."

[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

"These specifications apply both to the *spirits of* turpentine that is distilled from pine oleo-resins, commonly known as 'gum spirits' or 'spirits of turpentine,' and to turpentine commonly known as '*steam-distilled* wood turpentine' [which is obtained from resinous wood, whether by steam or by destructive distillation]. When ordering under these specifications, the purchaser shall specify whether (a) gum spirits *of turpentine* or (b) *steam-distilled* wood turpentine is desired. [When wood turpentine is specified, it may be stated whether steam or destructively distilled wood turpentine shall be furnished.]"

Section 6.—Under "Refractive index at 20° C." and under "Residue after Polymerization" change "Wood turpentine" to read "Steam-distilled wood turpentine."

STANDARD SPECIFICATIONS FOR BROKEN SLAG FOR WATERBOUND BASE AND WEARING COURSE

(SERIAL DESIGNATION: D 65 – 23)[1]

Section 4.—Omit this section, reading as follows, renumbering the succeeding sections accordingly:

"The percentage of wear shall be not more than 12.0 per cent."

Section 6.—Change the last line of this section to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"[Retained on] *Passing* 2½-in. screen . . . not [less] *more* than [85] *15* per cent."

Section 7 (b).—Omit this paragraph which refers to the method of testing the percentage of wear.

STANDARD SPECIFICATIONS FOR SHOVEL-RUN OR CRUSHER-RUN BROKEN SLAG FOR WATERBOUND BASE

(SERIAL DESIGNATION: D 66 – 23)[2]

Section 4.—Omit this section, reading as follows, renumbering the succeeding sections accordingly:

"The percentage of wear shall be not more than 15.0 per cent."

Section 6 (b).—Omit this paragraph which refers to the method of testing the percentage of wear.

[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

STANDARD METHOD OF TEST FOR DISTILLATION OF BITUMINOUS MATERIALS SUITABLE FOR ROAD TREATMENT

(SERIAL DESIGNATION: D 20 – 18)[1]

Section 4.—Change the first sentence to read as follows by the addition of the italicized word and the omission of the word in brackets:

"The apparatus shall be set up as shown in Fig. 2, the thermometer being placed so that the top of the bulb is opposite the *bottom* [middle] of the tubulature."

Make the position of the thermometer in the illustration correspond with this change.

Section 5.—Change the second sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"After adjusting the thermometer, shield, condenser, etc., the distillation is commenced, the rate being regulated that *50 to 70 drops* (*2.5 to 3.5 cc.*) *pass* [1 cc. passes] over every minute."

STANDARD METHODS OF LABORATORY SAMPLING AND ANALYSIS OF COKE

(SERIAL DESIGNATION: D 37 – 24)[2]

On page 1014 of the 1924 Book of A.S.T.M. Standards under "A Small Riffle Sampler," change "No. 10" to read "No. 20." Also change the paragraphs under "Roll Crusher" and under "Abbe Ball Mill or Hard-Steel Diamond Mortar" to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"*Hard-Steel* Roll Crusher.—For reducing the product passing a No. 4 sieve to pass a [No. 10] *No. 20* sieve."

"Abbe Ball Mill, *Hard-Steel Roll Crusher*, or Hard-Steel Diamond Mortar.—For reducing the product passing a [No. 10] *No. 20* sieve to pass a No. 60 sieve. The porcelain jars for the ball mill should be approximately 9 in. in diameter and 10 in. high. The flint pebbles should be smooth, hard, and well rounded. *The rolls of the hard-steel roll crusher should revolve at the same speed.*"

On page 1016 under "Reduction of Sample," line 9, change "No. 10 sieve" to read "No. 20 sieve;" also change "400 g." in lines 9 and 10 to read "200 g."

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. Prévost Hubbard, Secretary of Committee D-4 on Road and Paving Materials, 441 Lexington Ave., New York City.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. W. A. Selvig, Secretary of Committee D-5 on Coal and Coke, 4800 Forbes St., Pittsburgh, Pa.

Change the last two sentences of the first paragraph under "Reduction of Sample" from their present form: namely,

"Reduce the quantity of sample by quartering or riffling to about 50 g. Pass the entire 50-g. portion through a No. 60 sieve, pulverizing any course particles in a diamond mortar, and mix with remainder of sample, preserving the sample for analysis in a rubber-stoppered glass bottle."

to read as follows:

"Pass all the material through a No. 60 sieve, pulverizing any coarse particles by impact in a hard-steel diamond mortar, and mix with remainder of sample. Reduce the sample through small riffle sampler to about 50 g. and transfer to a rubber-stoppered glass bottle."

Change the last paragraph under "Reduction of Sample" to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"In case a ball mill is not available for fine grinding, quarter the 5-lb. sample passing a [No. 10] *No. 20* sieve to 200 g. and pulverize to pass a No. 60 sieve *by means of a hard-steel roll crusher in which the rolls revolve at the same speed, or* by impact in a hard-steel diamond mortar. The use of rubbing surfaces such as a disk pulverizer or a bucking board is never permissible for grinding coke."

STANDARD METHODS OF LABORATORY SAMPLING AND ANALYSIS OF COAL

(Serial Designation: D 22 – 24)[1]

On page 983 of the 1924 Book of A.S.T.M. Standards, under "Method of Sampling, (*A*) When Coal Appears Dry," line 12, change "120 g." to read "200 g."; also change "60 g." in line 6 to read "50 g." and on page 984, line 3, change "60 g." to read "about 50 g."

STANDARD METHODS OF SAMPLING AND ANALYSIS OF CREOSOTE OIL

(Serial Designation: D 38 – 24)[2]

A revision in the form of a separate tentative method entitled "Method of Test for Coke Residue of Creosote Oil (D 168 – 23 T)"[3] is intended to become a part of the present standard methods.

STANDARD METHODS OF TESTING MOLDED INSULATING MATERIALS

(Serial Designation: D 48 – 24)[4]

Section 2.—Change the first sentence to read as follows by the addition of the italicized words:

[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. W. A. Selvig, Secretary of Committee D-5 on Coal and Coke, 4800 Forbes St., Pittsbargh, Pa.

[2] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. J. A. Newlin, Secretary of Committee D-7 on Timber, Forest Products Laboratory, Madison, Wis.

[3] See p. 584.

[4] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. H. S. Vassar, Secretary of Committee D-9 on Electrical Insulating Materials, Twenty-first St. and Clinton Ave., Irvington, N. J.

"Any standard testing machine may be used *provided the error in the loading range does not exceed 1 per cent.*"

Section 3.—Change the second sentence to read as follows by the addition of the italicized word and the omission of those in brackets:

"It shall be molded in a hardened [and ground] steel mold *ground* to the dimensions given in Fig. 1."

Section 4.—Omit the second and third paragraphs reading as follows:

"Three specimens shall be tested after heating in an oven for one hour at a temperature which is 10° C. (18° F.) below the distortion point of the material, as determined in accordance with Sections 18 to 21. Each specimen shall be taken from the oven and tested immediately while hot.

"Two specimens shall be tested after they have been entirely immersed in water for 48 hours at normal room temperature. The specimens shall be pulled apart at normal room temperature of about 20° C. (68° F.) after the surface water has been removed by wiping with a dry cloth."

Change the first sentence of Paragraph (*b*) from its present form: namely,

"The test specimen shall be pulled apart at such a speed that the beam can be kept well balanced."

to read as follows:

"The speed of the head of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. (1.27 mm.) per minute with the machine running idle."

Fig. 2.—Change from a cube to a cylinder having a height of 1.25 in. (3.18 cm.) and a diameter of 1.125 in. (2.86 cm.). Omit the words "Manufacturer's Name and 'Test Specimen No. 3' Molded on Top in Small Round Body Raised Letters."

Section 6.—Change from its present form: namely,

"Any standard testing machine may be used. The pressure head used for standard compressive strength tests of cement blocks is satisfactory for this purpose. A sheet of soft annealed copper about $\frac{1}{32}$ in. (1 mm.) thick, or heavy blotting paper, shall be placed above and below the specimen to equalize irregularities."

to read as follows:

"Any standard testing machine may be used provided it is accurate to within 1 per cent for the lowest load for which it is used. One end of the specimen shall bear upon an accurately centered spherical bearing block, located whenever practicable at the top and the metal bearing plates shall be directly in contact with the ends of the test specimen. The pressure heads used for standard compression tests of cement (see Tentative Specifications and Tests

for Compressive Strength of Portland-Cement Mortars (Serial Designation: C 9 – 16 T) of the American Society for Testing Materials) are satisfactory for this test."

Section 7.—In the first sentence, change the word "cube" to "cylinder." Add a new sentence to read as follows:

"The ends shall be flat and perpendicular to the axis, being ground to this condition if necessary."

Section 8.—Omit the second and third paragraphs, reading as follows:

"Three specimens shall be crushed after heating for one hour at a temperature which is 10° C. (18° F.) below the distortion point of the material as determined in accordance with Sections 18 to 21. Each specimen shall be taken from the oven and tested immediately while hot.

"Two specimens shall be crushed after immersion in water at normal room temperature for 48 hours, with all surface water wiped off with a dry cloth."

Insert new paragraphs to read as follows:

"Additional tests shall be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each temperature selected. The specimen shall be kept at that temperature a sufficient length of time to become of uniform temperature throughout and shall be maintained at that temperature during the test.

"NOTE 1.—A convenient method of making this test is to submerge the specimens in a suitably lagged, electrically heated oil bath of about two gallons capacity. The specimens should be placed in the bath at least one-half hour before testing and the temperature maintained approximately constant at the desired testing temperature until the completion of that test. A pale mineral oil having a viscosity of 100 seconds ± 10 seconds at 38° C. (100° F.) Saybolt is satisfactory for this purpose. The specimens should not be placed in the oil an excessive time in advance of testing. The oil bath should be well stirred to insure uniform temperature throughout. The temperature should be determined by means of a thermocouple sealed in a similar specimen in the bath; or by the use of an A.S.T.M. partial immersion thermometer conforming to the Standard Specifications for Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F. (Serial Designation: D 182) of the American Society for Testing Materials,[1] so inserted in a hole in a specimen that the bulb is completely enclosed and sealed in.

"If tests are required over the whole range of temperature rather than at particular points, the method of testing may be found simpler and quicker if modified in accordance with Note 2, below.

"NOTE 2.—The oil with 5 specimens immersed in it should be heated to the highest temperature to be used and allowed to cool slowly. Specimens should be tested in succession and the average temperature during the actual time of testing of each specimen taken as that corresponding to each strength deter-

[1] A.S.T.M. Standards Adopted in 1925.

mination. Specimens should be added to the bath successively on such a schedule that each will remain in the bath at least ½ hour before testing. An average curve shall be drawn through the plotted data."

Change the first sentence of Paragraph (*b*) from its present form: namely,

"The load shall be applied in a direction at right angles to that in which the pressure was applied in molding, and at such a rate of speed that will permit the beam to be kept well balanced from zero load until the specimen is crushed."

to read as follows:

"The load shall be applied on the ends of the specimen and the cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle."

In Paragraph (*e*), change the word "jaws" to read "head." Add a new sentence to read as follows:

"The time required for testing each specimen should be recorded."

Section 10.—Change to read as follows:

"Any standard testing machine may be used provided it is accurate to within 1 per cent for the lowest load for which it is used. The distance between points of support shall be 4 in. (10.16 cm.). The supports shall have the contact edges rounded to a radius of ⅛ in. (3.18 mm.). The load shall be applied midway between the supports by a pressure piece, the bearing edge of which shall be rounded to a radius of ⅛ in. (3.18 mm.)."

Section 12.—In the first sentence of Paragraph (*a*) change the words "four specimens" to read "five specimens."

Change the second paragraph from its present form: namely,

"Three specimens shall be tested after immersion in water at normal room temperature for 48 hours with all surface water wiped off with a dry cloth."

to read as follows:

"Additional tests shall be made at elevated temperatures as outlined in Section 8."

Change the first two sentences of Paragraph (*b*) from their present form: namely,

"The load shall be applied at as slow a speed as possible, so that the beam may be kept well balanced from zero load until the first sign of failure. All tests shall be made at room temperature of about 20° C. (68° F.)."

to read as follows:

"The cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle."

Section 13.—Change Paragraph (*e*) to read as follows by the addition of the italicized word and the omission of those in brackets:

"The [amount of] *maximum* deflection at the center in inches or in millimeters."

Omit Paragraph (*f*) reading as follows:

"The distance between the supports in inches or in millimeters."

Section 16 (*b*).—Change the second paragraph from its present form: namely,

"Three specimens shall be punctured after heating one hour in an oven heated to 10° C. (18° F.) below the distortion point of the material as determined in Sections 18 to 21. The specimen shall be taken from the oven and immediately punctured in air while hot. This test is intended to cover all molded materials which do not withstand working temperatures above 125° C. (257° F.). Tests on materials which resist high temperatures may be made above 125° C. (257° F.) when required."

to read as follows:

"Additional tests shall be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each temperature while in an oven maintained at the proper temperature, either by hand adjustment or by automatic thermostat. Specimens shall be placed in the oven at least ½ hour previous to testing and the temperature shall then be maintained approximately constant at the testing temperature until the completion of that test. Temperature shall be determined by means of a thermocouple or a mercury thermometer placed in the oven close to the specimens and care shall be taken to avoid placing the specimens too close to the heater.

"Specimens with which arcing over the edge is encountered shall be tested under oil as described under Section 16, and in such cases the oil bath containing the specimens shall be installed in the oven and the temperature determined by immersing the thermometer in the oil."

In the third paragraph, change the words "Two specimens" to "Three specimens."

Section 17.—Add a new paragraph to read as follows:

"(*e*) Temperature at which each test was made."

Section 23.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

"The test specimen [No. 1] *No. 3* shown in [Fig. 4] *Fig. 2* shall be used for this test. [After the puncture tests prescribed in Section 16 (*b*) are completed on the five specimens, three of them shall be taken and the entire rim of each specimen shall be sawed off with a hack saw ¼ in. (6 mm.) back from the top edge of the specimen so as to expose a more or less uniformly cut surface.]"

Section 24.—Change the first sentence from its present form: namely,

"Weigh each of the three test specimens after the rim has been sawed off."

to read as follows:

"Three specimens shall be tested. Weigh each of the three separately in the as-received condition."

In the fifth and sixth sentences change the words "100 hours" to "48 hours."

Section 25.—In paragraphs (*c*) and (*d*) change the words "100 hours" to " 48 hours."

STANDARD DEFINITIONS OF TERMS RELATING TO PAINT SPECIFICATIONS

(SERIAL DESIGNATION: D 16 – 24)[1]

Add the following three new definitions of terms:

Toughness.—Relative degree of resistance to impact without fracture; apposed to brittleness.

Elasticity of Paint and Varnish Films.—That property which allows a paint or varnish film to follow, without rupture, changes in the extent and form of the surface to which it is applied.

"*Screen* (*Sieve*).—A plate or sheet or woven cloth with regularly spaced apertures of uniform size, mounted on a suitable frame or holder, for use in separating materials according to size."

"NOTE.—The shape and spacing of apertures, size of wires or threads, thickness of plate or sheet, allowable variations and similar properties should be described in the specifications."

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[1] 1924 Book of A.S.T.M. Standards. Criticisms of this revision are solicited and should be directed to Mr. R. L. Hallett, Secretary of Committee D-1 on Preservative Coatings for Structural Materials, 105 York St., Brooklyn, N. Y.

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LIST OF STANDARDS[1]

Published Collectively in their latest revised form in 1924 BOOK OF A.S.T.M. STANDARDS with 1925 Supplement to the BOOK OF STANDARDS

The designations A 1, A 2, etc., of the Standards are fixed; the final numbers 14, 24, etc., indicate the year of adoption as standard or, in the case of revision, the year of last revision. The serial designations of Standards, which have for any reason been discontinued, are permanently dropped. In ordering Standards, the complete serial designations should be stated, thus, A 1 – 24, A 2 – 24, etc.

A. FERROUS METALS

STEEL

See also **WROUGHT IRON:** A 56 – 24

STANDARD SPECIFICATIONS

Steel Rails and Accessories.

A 1 – 24. For Carbon-Steel Rails.
A 2 – 24. For the Manufacture of Open-hearth Steel Girder Rails of Plain, Grooved and Guard Types.
A 3 – 24. For Low-Carbon-Steel Splice Bars.
A 4 – 14. For Medium-Carbon-Steel Splice Bars.
A 5 – 14. For High-Carbon-Steel Splice Bars.
A 6 – 14. For Extra-High-Carbon-Steel Splice Bars.
A 49 – 21. For Quenched High-Carbon-Steel Splice Bars.
A 50 – 24. For Quenched Carbon-Steel Track Bolts.
A 51 – 21. For Quenched Alloy-Steel Track Bolts.
A 65 – 24. For Steel Track Spikes.
A 66 – 21. For Steel Screw Spikes.

Structural Steel.

A 7 – 24. For Structural Steel for Bridges.
A 8 – 24. For Structural Nickel Steel.
A 9 – 24. For Structural Steel for Buildings.
A 10 – 24. For Structural Steel for Locomotives.
A 11 – 24. For Structural Steel for Cars.
A 12 – 21. For Structural Steel for Ships.
A 13 – 24. For Rivet Steel for Ships.

[1] As of August 28, 1925. Total number, 254.

Spring Steel and Springs.

A 14 – 16. For Carbon Steel-Bars for Railway Springs.
A 68 – 18. For Carbon-Steel Bars for Railway Springs with Special Silicon Requirements.
A 58 – 16. For Carbon-Steel Bars for Vehicle and Automobile Springs.
A 59 – 16. For Silico-Manganese-Steel Bars for Automobile and Railway Springs
A 60 – 16. For Chrome-Vanadium-Steel Bars for Automobile and Railway Springs.
A 61 – 16. For Helical Steel Springs for Railways.
A 62 – 16. For Elliptical Steel Springs for Railways.
A 69 – 18. For Elliptical Steel Springs for Automobiles.

Reinforcement Bars.

A 15 – 14. For Billet-Steel Concrete Reinforcement Bars.
A 16 – 14. For Rail-Steel Concrete Reinforcement Bars.

Steel Blooms, Forgings and Axles.

*A 17 – 21. For Carbon-Steel and Alloy-Steel Blooms, Billets and Slabs for Forgings.
*A 18 – 21. For Carbon-Steel and Alloy-Steel Forgings.
A 19 – 21. For Quenched-and-Tempered Carbon-Steel Axles, Shafts and Other Forgings for Locomotives and Cars.
A 63 – 21. For Quenched-and-Tempered Alloy-Steel Axles, Shafts, and Other Forgings for Locomotives and Cars.
A 20 – 21. For Carbon-Steel Forgings for Locomotives.
A 21 – 18. For Carbon-Steel Car and Tender Axles.
A 22 – 21. For Cold-Rolled Steel Axles.

Steel Wheels and Tires.

A 25 – 24. For Wrought Solid Carbon-Steel Wheels for Electric Railway Service.
A 57 – 24. For Wrought Solid Carbon-Steel Wheels for Steam Railway Service.
A 26 – 16. For Steel Tires.

Steel Castings.

A 27 – 24. For Steel Castings.

Steel Tubes and Pipe.

A 83 – 24. For Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes.
A 53 – 24. For Welded and Seamless Steel Pipes.

Automotive Steels (see also **Spring Steel**).

A 29 – 24. For Automotive Carbon and Alloy Steels.

Boiler Steels.

A 30 – 24. For Boiler and Firebox Steel for Locomotives.
A 70 – 24. For Boiler and Firebox Steel for Stationary Service.
A 31 – 24. For Boiler Rivet Steel.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

Bar Steels.

A 32 – 24. For Cold-Finished Bessemer Steel Automatic Screw Stock.
A 54 – 24. For Cold-Finished Open-Hearth Steel Automatic Screw Stock.
A 80 – 24. For Commercial Bar Steels.

STANDARD METHODS

A 34 – 24. Of Test for Magnetic Properties of Iron and Steel.
A 33 – 24. Of Chemical Analysis of Plain Carbon Steel.
A 55 – 24. Of Chemical Analysis of Alloy Steels.
A 90 – 24. Of Determining Weight of Coating on Zinc-Coated Articles.
A 91 – 24. Of Determining Weight of Coating on Tin, Terne, and Lead-Coated Sheets.

RECOMMENDED PRACTICE

A 35 – 24. For Annealing of Miscellaneous Rolled and Forged Carbon-Steel Objects.
A 36 – 24. For Heat Treatment of Carbon-Steel Castings.
A 37 – 14. For Heat Treatment of Case-Hardened Carbon-Steel Objects.

WROUGHT IRON

(See also **STEEL:** A 83 – 24)

STANDARD SPECIFICATIONS

A 72 – 24. For Welded Wrought-Iron Pipe.
A 84 – 24. For Staybolt, Engine-Bolt and Extra-Refined Wrought-Iron Bars.
*A 41 – 18. For Refined Wrought-Iron Bars.
A 85 – 24. For Merchant Bar Iron.
*A 42 – 18. For Wrought-Iron Plates.
A 73 – 24. For Wrought-Iron Rolled or Forged Blooms and Forgings for Locomotives and Cars.
A 56 – 24. For Iron and Steel Chain.

STANDARD DEFINITIONS

A 81 – 21. Of Terms Relating to Wrought-Iron Specifications.

PIG IRON, CAST IRON, AND FINISHED CASTINGS

STANDARD SPECIFICATIONS

A 43 – 24. For Foundry Pig Iron.
A 44 – 04. For Cast-Iron Pipe and Special Castings.
A 74 – 18. For Cast-Iron Soil Pipe and Fittings.
A 45 – 14. For Cast-Iron Locomotive Cylinders.
A 46 – 24. For Chilled Cast-Iron Wheels.
A 47 – 24. For Malleable Castings.
A 48 – 18. For Gray-Iron Castings.
A 88 – 24. For High-Test Gray-Iron Castings.

STANDARD METHODS

A 64 – 16. Of Sampling and Chemical Analysis of Pig and Cast Iron.

*Approved as "Tentative American Standard" by the American Engineering Standards Committee.

B. NON-FERROUS METALS

STANDARD SPECIFICATIONS

Ingot Copper.

*B 4 – 13. For Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars.

*B 5 – 13. For Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars.

Spelter.

B 6 – 18. For Spelter.

Lead.

B 29 – 23. For Pig Lead.

Nickel.

B 39 – 22. For Nickel.

Aluminum Alloys.

B 26 – 21. For Light Aluminum Casting Alloys.

Wire and Cable.

B 1 – 23. For Hard-Drawn Copper Wire.

B 2 – 15. For Medium Hard-Drawn Copper Wire.

*B 3 – 15. For Soft or Annealed Copper Wire.

B 33 – 21. For Tinned Soft or Annealed Copper Wire for Rubber Insulation.

B 8 – 21. For Bare Concentric-Lay Copper Cable: Hard, Medium-Hard, or Soft.

B 47 – 25. For Round and Grooved Hard-Drawn Copper Trolley Wire.

B 9 – 16. For High-Strength Bronze Trolley Wire, Round and Grooved: 40 and 65-per-cent Conductivity.

Bronze.

B 10 – 18. For the Alloy: Copper, 88 per cent; Tin, 10 per cent; Zinc, 2 per cent.

*B 30 – 22. For Brass Ingot Metal, Graded and Ungraded, for Sand Castings.

B 31 – 21. For Bronze Bearing Metal in Ingot Form.

B 22 – 21. For Bronze Bearing Metals for Turntables and Movable Railroad Bridges.

Solder Metal.

*B 32 – 21. For Solder Metal.

Copper and Brass Plates, Tubes, Rods, etc.

B 11 – 18. For Copper Plates for Locomotive Fireboxes.

B 12 – 21. For Copper Bars for Locomotives Staybolts.

B 13 – 18. For Seamless Copper Boiler Tubes.

B 14 – 18. For Seamless Brass Boiler Tubes.

B 44 – 24. For Seamless Admiralty Condenser Tubes and Ferrule Stock.

B 55 – 25. For Seamless 70–30 Brass Condenser Tubes and Ferrule Stock.

B 56 – 25. For Seamless Muntz Metal Condenser Tubes and Ferrule Stock.

B 42 – 24. For Copper Pipe, Standard Sizes.

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* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

Copper and Brass Plates, Tubes, Rods, etc.—**Continued.**

B 43 – 24. For Brass Pipe, Standard Sizes.
*B 15 – 18. For Brass Forging Rod.
*B 16 – 18. For Free-Cutting Brass Rod for Use in Screw Machines.
B 19 – 19. For Cartridge Brass.
B 20 – 19. For Cartridge Brass Disks.
*B 21 – 19. For Naval Brass Rods for Structural Purposes.
*B 36 – 21. For High Sheet Brass.

STANDARD METHODS

*B 34 – 20. Of Battery Assay of Copper.
B 35 – 24. Of Chemical Analysis of Pig Lead.
B 38 – 21. Of Chemical Analysis of Spelter.
B 41 – 23. Of Chemical Analysis of Nickel.
*B 18 – 21. Of Chemical Analysis of Alloys of Lead, Tin, Antimony and Copper.
*B 27 – 19. Of Chemical Analysis of Manganese Bronze
*B 28 – 19. Of Chemical Analysis of Gun Metal.
B 45 – 23. Of Chemical Analysis of Brass Ingots and Sand Castings.
B 46 – 23. Of Chemical Analysis of Bronze Bearing Metal.

C. CEMENT, LIME, GYPSUM, AND CLAY PRODUCTS

STANDARD SPECIFICATIONS

†C 9 – 21. Specifications and Tests for Portland Cement.
C 10 – 09. For Natural Cement.
C 7 – 15. For Paving Brick.
C 21 – 20. For Building Brick.
C 32 – 24. For Clay Sewer Brick.
C 13 – 24. For Clay Sewer Pipe.
C 14 – 24. For Cement-Concrete Sewer Pipe.
C 4 – 24. For Drain Tile.
C 6 – 24. For Hydrated Lime for Structural Purposes.
C 45 – 25. For Quicklime and Hydrated Lime for Use in the Cooking of Rags for the Manufacture of Paper.
C 48 – 24. For Quicklime and Hydrated Lime for Use in the Textile Industry.
C 49 – 24. For Quicklime and Hydrated Lime for the Manufacture of Silica Brick.
C 22 – 25. For Gypsum.
C 23 – 22. For Calcined Gypsum.
C 28 – 21. For Gypsum Plasters.
C 35 – 25. For Gypsum Plastering Sand.
C 36 – 25. For Gypsum Wall Board.
C 37 – 25. For Gypsum Plaster Board.
C 52 – 25. For Gypsum Partition Tile or Block.
*C 19 – 18. For Fire Tests of Materials and Construction.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.
† Approved as "American Standard" by the American Engineering Standards Committee.

Standard Methods

C 16 – 20. Of Test for Refractory Materials under Load at High Temperatures.
C 20 – 20. Of Test for Porosity and Permanent Volume Changes in Refractory Materials.
C 24 – 20. Of Test for Softening Point of Fire-Clay Brick.
C 18 – 21. Of Ultimate Chemical Analysis of Refractory Materials, Including Chrome Ores and Chrome Brick.
*C 29 – 21. Of Test for Unit Weight of Aggregate for Concrete.
*C 30 – 22. Of Test for Voids in Fine Aggregate for Concrete.
C 31 – 21. Of Making and Storing Specimens of Concrete in the Field.
C 39 – 25. Of Making Compression Tests of Concrete.
*C 40 – 22. Of Test for Organic Impurities in Sands for Concrete.
C 41 – 24. Of Test for Sieve Analysis of Aggregates for Concrete.
C 26 – 23. Of Testing Gypsum and Gypsum Products.

Standard Definitions

C 8 – 24. Of Terms Relating to Sewer Pipe.
C 27 – 20. For Clay Refractories.
C 43 – 24. Of Terms Relating to Hollow Tile.

Recommended Practice

C 12 – 19. For Laying Sewer Pipe.

D. MISCELLANEOUS MATERIALS

PRESERVATIVE COATINGS

Standard Specifications

D 1 – 15. For Purity of Raw Linseed Oil from North American Seed.
D 11 – 15. For Purity of Boiled Linseed Oil from North American Seed.
D 13 – 24. For Turpentine.
D 125 – 23. For Perilla Oil, Raw or Refined.
D 79 – 24. For Zinc Oxide.
D 80 – 24. For Leaded Zinc Oxide.
D 81 – 24. For Basic Carbonate White Lead.
D 82 – 24. For Basic Sulfate White Lead.
D 83 – 24. For Red Lead.
D 85 – 24. For Ocher.

Standard Methods

D 153 – 24. Of Test for Specific Gravity of Pigments.
D 154 – 24. Of Testing Oleo-Resinous Varnishes.
D 154 – 25. Of Testing Oleo-Resinous Varnishes.[1]
D 29 – 24. Of Testing Shellac.
D 29 – 25. Of Test for Determination of Wax in Shellac.[2]
*D 56 – 21. Of Test for Flash Point of Volatile Flammable Liquids.
*D 34 – 17. Of Routine Analysis of White Pigments.
*D 49 – 18. Of Routine Analysis of Dry Red Lead.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

[1] To be added to the Standard Methods of Testing Oleo-Resinous Varnishes (Serial Designation: D 154 – 24) when the Book of Standards is next published.

[2] To be added to the present Standard Methods of Testing Shellac (Serial Designation: D 29 – 24) when the Book of Standards is next published.

*D 50 – 18. Of Routine Analysis of Yellow, Orange, Red and Brown Pigments Containing Iron and Manganese.
D 126 – 23. Of Routine Analysis of Yellow and Orange Pigments Containing Chromium Compounds, Blue Pigments and Chrome Green.
D 186 – 25. Of Routine Analysis of Titanium Pigments.

STANDARD DEFINITIONS

D 16 – 24. Of Terms Relating to Paint Specifications.

PETROLEUM PRODUCTS AND LUBRICANTS

STANDARD METHODS

D 47 – 24. Of Test for Specific Gravity of Lubricants.
D 206 – 25. Abridged Volume Correction Table for Petroleum Oils.
D 87 – 22. Of Test for Melting Point of Paraffin Wax.
D 92 – 24. Of Test for Flash and Fire Points by Means of Open Cup.
D 93 – 22. Of Test for Flash Point by Means of the Pensky-Martens Closed Tester.
D 95 – 24. Of Test for Water in Petroleum Products and Other Bituminous Materials.
D 96 – 24. Of Test for Water and Sediment in Petroleum Products by Means of Centrifuge.

ROAD MATERIALS

STANDARD SPECIFICATIONS

*D 57 – 20. For Materials for Cement Grout Filler for Brick and Stone Block Pavements.
D 58 – 24. For Materials for Cement Mortar Bed for Brick, Stone Block, Wood Block, Asphalt Block and Other Block Pavements.
*D 59 – 22. For Block for Granite Block Pavements.
D 131 – 23. For Block for Recut Granite Block Pavements.
D 132 – 23. For Block for Durax Granite Pavements.
D 65 – 23. For Broken Slag for Waterbound Base and Wearing Course.
D 66 – 23. For Shovel-Run or Crusher-Run Broken Slag for Waterbound Base.

STANDARD METHODS

D 2 – 08. Of Test for Abrasion of Road Material.
*D 3 – 18. Of Test for Toughness of Rock.
*D 30 – 18. Of Test for Apparent Specific Gravity of Course Aggregates.
D 55 – 25. Of Test for Apparent Specific Gravity of Sand, Stone and Slag Screenings, and Other Fine Non-Bituminous Highway Materials.
D 72 – 21. Of Test for Quantity of Clay and Silt in Gravel for Highway Construction.
*D 75 – 22. Of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

D 7 – 18. Of Mechanical Analysis of Sand or Other Fine Highway Material, except Fine Aggregates Used in Cement Concrete.
D 18 – 16. Of Mechanical Analysis of Broken Stone or Broken Slag, except Aggregates Used in Cement Concrete.
D 19 – 16. Of Mechanical Analysis of Mixtures of Sand or Other Fine Material with Broken Stone or Broken Slag, except Aggregates Used in Cement Concrete.
D 35 – 18. Form of Specifications for Certain Commercial Grades of Broken Stone.
D 5 – 25. Of Test for Penetration of Bituminous Materials.
D 6 – 20. Of Test for Loss on Heating of Oil and Asphaltic Compounds.
*D 20 – 18. Of Test for Distillation of Bituminous Materials Suitable for Road Treatment.
D 36 – 24. Of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method).
D 61 – 24. Of Test for Softening Point of Tar Products (Cube-in-Water Method).
D 140 – 25. Of Sampling Bituminous Materials.

Standard Definitions

D 8 – 18. Of Terms Relating to Materials for Roads and Pavements.

COAL AND COKE

Standard Specifications

D 17 – 16. For Foundry Coke.
D 166 – 24. For Gas and Coking Coals.

Standard Methods

*D 21 – 16. Of Sampling Coal.
D 22 – 24. Of Laboratory Sampling and Analysis of Coal.
D 37 – 24. Of Laboratory Sampling and Analysis of Coke.
D 141 – 23. Of Shatter Test for Coke.
D 167 – 24. Of Test for Volume of Cell Space of Lump Coke.

TIMBER AND TIMBER PRESERVATIVES

Standard Specifications

D 10 – 15. For Yellow-Pine Bridge and Trestle Timbers.
D 24 – 20. For Southern Yellow-Pine Timber to be Creosoted.
D 25 – 20. For Southern Yellow-Pine Piles and Poles to be Creosoted.
D 52 – 20. For Wooden Paving Blocks for Exposed Pavements.

Standard Methods

D 38 – 24. Of Sampling and Analysis of Creosote Oil.

Standard Definitions

D 9 – 15. Of Terms Relating to Structural Timber.

* Approved as "Tentative American Standard" by the American Engineering Standards Committee.

WATERPROOFING

STANDARD SPECIFICATIONS

D 40 – 25. For Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level.
D 144 – 25. For Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level.
D 41 – 25. For Primer for Use with Asphalt in Damp-proofing and Waterproofing Below and Above Ground Level.
D 42 – 25. For High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level.
D 145 - 25. For High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level.
D 200 – 25. For High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level.
D 201 – 25. For High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level.
D 43 – 25. For Creosote Oil for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level.
D 169 – 25. For Ashpalt Mastic for Use in Waterproofing.
D 170 – 25. For Bituminous Grout for Use in Waterproofing Above Ground Level.
D 171 – 25. For Bituminous Grout for Use in Waterproofing Below Ground Level.
D 173 – 25. For Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing.
D 174 – 25. For Burlap Saturated with Bituminous Substances for Use in Waterproofing.

INSULATING MATERIALS

STANDARD METHODS

D 48 – 24. Of Testing Molded Insulating Materials.
D 117 – 24. Of Testing Transformer and Switch Oils.

RUBBER PRODUCTS

STANDARD SPECIFICATIONS

D 14 – 23. For Cotton Rubber-Lined Fire Hose for Private Fire Department Use.
D 26 – 23. For Cotton Rubber-Lined Fire Hose for Public Fire Department Use.
D 46 – 24. For Wrapped Air Hose for Use with Pneumatic Tools.
D 60 – 24. For Braided Air Hose for Use with Pneumatic Tools.
D 177 – 24. For Wrapped Cold Water Hose.
D 53 – 24. For Rubber Belting for Power Transmission.
D 120 – 23. For Rubber Gloves for Electric Workers on Apparatus or Circuits Not Exceeding 3000 Volts to Ground.

D 178 – 24. For Rubber Matting for Use Around Electrical Apparatus or Circuits Not Exceeding 3000 Volts to Ground.
D 151 – 23. For Rubber Pump Valves.
D 69 – 24. For Adhesive Tape for General Use for Electrical Purposes.

Standard Methods

D 15 – 24. Of Testing Rubber Products.

TEXTILE MATERIALS

Standard Specifications

D 122 – 25. For Imperfections and Tolerances for Square-Woven Tire Fabrics.
D 179 – 25. For Imperfections and Tolerances for Cord Tire Fabrics.
D 181 – 25. For Tolerances for Hose Ducks and Belt Ducks.
D 203 – 25. For Tolerances and Test Methods for Electrical Cotton Yarns.
D 205 – 25. Specifications and Tests for Osnaburg Cement Sacks.

Standard Methods

D 39 – 24. Of Testing Cotton Fabrics.

THERMOMETERS

Standard Specifications

D 182 – 25. For A.S.T.M. Partial-Immersion Thermometer for General Use, -20 to $+150°$ C., 0 to $+300°$ F.
D 183 – 25. For A.S.T.M. Partial-Immersion Thermometer for General Use, -5 to $+300°$ C., $+20$ to $+580°$ F.
D 184 – 25. For A.S.T.M. Partial-Immersion Thermometer for General Use, -5 to $+400°$ C., $+20$ to $+760°$ F.

E. MISCELLANEOUS SUBJECTS

Standard Methods

E 4 – 24. Of Verification of Testing Machines.
E 3 – 24. Of Metallographic Testing of Iron and Steel.
E 5 – 24. Of Metallographic Testing of Non-Ferrous Metals and Alloys.

Standard Rules

E 2 – 24. Governing the Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography.

Standard Definitions

E 7 – 24. Of Terms Relating to Metallography.

PRICE LIST OF PUBLICATIONS

(August 28, 1925)

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